

# **Surface Water, Groundwater, and Solid Phase Media Investigation Work Plan - Mayflower Mill and Tailings Impoundments Area**

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## LIST OF ACRONYMS

ARSG	Animas River Stakeholder Group
DQO	Data Quality Objective
EPA	United States Environmental Protection Agency
HASP	Health and Safety Plan
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
TA	Target Analyte



## 1.0 INTRODUCTION

This Surface Water, Groundwater, and Solid Phase Media Investigation Work Plan (Work Plan) was prepared on behalf of Sunnyside Gold Corporation (Sunnyside) to investigate the surface water, groundwater, and solid phase media in the Upper Animas River Valley in the area at and adjacent to the Mayflower Mill and Tailings Impoundments Area near Silverton, Colorado (Figure 1-1). The Work Plan describes the sampling and analysis plan (SAP) for the multi-media sampling events planned for the summer and fall of 2015. A subsurface investigation work plan has been prepared as a separate document that describes drilling, core sampling, and monitor well construction on the in the Mayflower Mill and Tailings Impoundments Area planned for the summer of 2015 (Formation Environmental, 2015a).

There are four Mayflower Tailings Impoundments located approximately one mile to the northeast and upstream of Silverton on the right bank of the Animas River, as shown on Figure 1-2. The study area in the upper Animas River Valley extends along the river and the floodplain from just upstream of the confluence of the upper Animas River and Arrastra Creek downstream to the 14<sup>th</sup> Street bridge crossing in Silverton. The focus of this subsurface investigation is the Mayflower Mill and Tailings Impoundment Area. Additional locations outside of this study area may also be investigated, as directed by Sunnyside. Investigations in these areas would be conducted in accordance with this Work Plan.

The upper Animas River basin and its tributaries are intensely mineralized, and natural weathering of mineralized rock degrades the basin's surface water quality. Streams within the basin that are considered representative of natural-background conditions (i.e., unaffected or minimally affected by mining activity) can be acidic (pH < 3.0) with trace metals concentrations, including zinc, copper, and manganese, above aquatic life standards (USGS, 2007). Environmental conditions in the upper Animas River basin also reflect influences from the extensive historic mining and milling activities that occurred over the past 150 years, including mining in areas upstream of the Mayflower Mill and Tailings Impoundments Area and on the left bank of the Animas River (the opposite bank from the Mayflower Mill and Tailings Impoundments Area). Mine adits and historic mine waste rock piles are present at numerous locations, and historic mills typically discharged tailings to the Animas and its tributaries.

Aside from this introductory section, the structure of this Work Plan is as follows. A discussion of the background information is presented in Section 2.0. Section 3.0 identifies the data needs, intended data uses, and data quality objectives (DQOs) for the investigation. The SAP for surface water, pore water, groundwater, sediment, and opportunistic sampling is presented in Section 4.0 along with a list of Target Analytes (TAs). Reporting of the results for the multi-media sampling effort is described in Section 5.0. References cited in this Work Plan are listed in Section 6.0. Appendices to this Work Plan are listed below:

- Appendix A: Quality Assurance Project Plan (QAPP)
- Appendix B: Standard Operating Procedures (SOP)
- Appendix C: Health and Safety Plan (HASP)

## 2.0 BACKGROUND

Background information is described in the Subsurface Investigation Work Plan (Formation Environmental, 2015a) and the High-Flow Surface Water Investigation Work Plan (Formation Environmental, 2015b).

### **3.0 DATA QUALITY OBJECTIVES AND DATA NEEDS**

This section identifies the data needs, intended data uses, and DQOs for the multi-media investigation of the study area shown by Figure 1-2. The target analytes (TAs) for the multi-media investigation are described in Section 4.0.

This section is organized to be generally consistent with Environmental Protection Agency's (EPA's) guidance for application of its DQO process (EPA, 2006), which includes the seven steps listed below.

1. State the Problem
2. Identify the Goals of the Study
3. Identify Information Inputs
4. Define the Boundaries of the Study
5. Develop the Analytic Approach
6. Specify Performance and Acceptance Criteria
7. Develop the Plan for Collecting Data

Application of the DQO process results in identification of the specific types and quality of data needed to support the goals of the surface water investigation.

The DQOs listed below are in part based on the EPA SAP/QAPP for the Upper Animas Mining District (EPA 2015a) and, where appropriate, additional DQOs have been added to meet the specific objectives for the investigation of the study area.

#### **3.1 DQO Step 1 - State the Problem**

Previous investigations in the study area have identified elevated levels of metals in the waters of the upper Animas River in the vicinity of the Mayflower Mill and Tailings Impoundments Area. The materials present in the tailings impoundments may or may not be sources of metals to the river via leaching and subsurface transport by groundwater. However, the current sources of metals and their effects on river water quality remain uncertain. Therefore, additional data are needed to better understand the relationship, if any, between the Mayflower Mill and Tailings Impoundments Area and metals concentrations in surface water of the upper Animas River adjacent to and downstream from this area. More specifically, additional physical and chemical data are needed to evaluate the metals concentrations in the surface water of the upper Animas River and in groundwater at and downgradient from this area as well as left bank sources. Characterization of metals concentrations in nearby Animas River sediments and sediment pore water and soil at selected locations is also needed to better understand the nature and extent of metals contamination within the study area. Additional areas outside of the

study area may be investigated, as directed by Sunnyside. Investigations in these areas would be conducted in accordance with this Work Plan.

### **3.2 DQO Step 2 - Identify the Goals of the Study**

The key questions to be answered by the multi-media investigation are as follows:

- How do the concentrations of the TAs change in surface water in response to varying flow conditions (e.g., high flow vs. low flow)?
- How do the concentrations of the TAs change in surface water with location along the reach of interest in the study area?
- How do the concentrations of the TAs change in surface water in relation to known inflows along the reach of interest in the study area?
- What are the concentrations of the TAs in groundwater at and upgradient from the Mayflower Mill and Tailings Impoundments Area and how do concentrations vary with location and depth?
- What are the groundwater conditions (i.e., water bearing zones, depth, flow direction, and hydraulic gradient) in the Mayflower Mill and Tailings Impoundments Area?
- What is the current nature and extent of metals contamination in the Animas River sediments and pore water and how do the concentrations change with location along the reach of interest in the study area?
- What is the nature and extent of metals contamination in soil, mine waste, and tailings in selected locations within the study area?

### **3.3 DQO Step 3 - Identify Information Inputs**

Previous investigations provided preliminary characterization of the surface water flow conditions and surface water quality in the study area in 2002 and 2003 (Kimball et al., 2010). However, since then there have been remedial actions along the Animas River, and the available data may no longer be representative of current conditions. Additionally, there are significant data gaps in the characterization of the groundwater and solid-phase media in the study area. As such, the following types of information are needed to address the study goals listed in DQO Step 2.

- Concentrations of the surface water TAs at multiple locations on the upper Animas River reach of interest, including locations of inflowing water along that reach, during both relatively high-flow and low-flow conditions.
- Concentrations of sediment and pore water TAs at multiple locations on the upper Animas River reach of interest.
- Concentrations of groundwater TAs at multiple locations within study area.
- Concentrations of TAs in soil and mine wastes and the leaching potential of these solid-phase media at multiple locations within the study area.
- The available flow and water chemistry data collected by others for the upper Animas River.
- River discharge records for years 2002 through 2015 from the USGS Animas River gauging station 0935800.
- Depth to groundwater measurements at monitoring wells located in the study area.
- Field water quality parameters, observations, and measurements.

### **3.4 DQO Step 4 – Define the Boundaries of the Study**

The spatial boundaries for the study area are shown on Figure 1-2 and extend along the river and the floodplain from just upstream of the confluence of the upper Animas River and Arrastra Creek downstream to the 14<sup>th</sup> Street bridge crossing. Additional areas outside of the study area may be investigated, as directed by Sunnyside. Investigations in these areas would be conducted in accordance with this Work Plan.

The temporal boundaries for the study begin in 2002 with the low-flow sampling event conducted by the USGS and end with the final sampling event proposed for the summer/fall of 2015.

### **3.5 DQO Step 5 – Develop the Analytic Approach**

The following approach will be used to collect the specific types of new data needed to address the goals of the multi-media investigation. Two or more surface water synoptic sampling events are to be conducted within the reach of the upper Animas River in the study area. The events are to be conducted at relatively high-flow (sampling event was conducted in May 2015 in accordance with the High-Flow Surface Water Investigation Work Plan; Formation, 2015b) and low-flow conditions. Additionally, where feasible, the major inflows in the study area that were

identified by the USGS during its 2002 study, including tributary creeks, streams, and seeps, will be sampled.). Selected groundwater monitoring wells will be sampled one or more times. Sediment and sediment pore water will be sampled one or more times in the upper Animas River. Various solid phase media (e.g., soil, tailings, mine waste, mineralized rock, etc.) will be sampled at selected locations in the study area.

The data analyses that will be performed as part of the multi-media investigation are as follows:

1. Compare the high-flow surface water monitoring results to the low-flow monitoring results. Evaluate and describe the observed differences in TA concentrations for the two distinct flow conditions.
2. Compare the upper Animas River data collected during this investigation to historical data published by others (e.g., USGS, Animas River Stake Holder Group [ARSG], and EPA).
3. Use depth-to-water data to determine groundwater flow direction(s) and hydraulic gradient(s) within the study area.
4. Evaluate the concentrations of TAs in groundwater and their spatial distribution. Develop isoconcentration contour maps for selected dissolved phase chemicals of concern, if appropriate.
5. Compare surface water and groundwater results.

Measurement errors (field and laboratory) and related uncertainties for the data collected and the results of the data-analyses described above will be evaluated and described.

### **3.6 DQO Step 6 – Specify Performance or Acceptance Criteria**

Performance and acceptance criteria are defined and controlled through implementation of sampling and analytical methodologies that are designed to ensure that the data generated are of adequate quality for project decision-making purposes. If the quality assurance activities for sample collection and analysis specified in project documents are met, and the analytical precision and accuracy requirements specified in the QAPP (Appendix A) are met, the resulting data will be usable for characterizing the conditions in the study area and addressing the study goals stated herein.

The laboratory analysis methods need to provide quantitative data at concentrations low enough for meaningful comparison to applicable regulatory standards and/or thresholds (e.g., surface water quality standards, etc.). The proposed analytical methods and the target method detection

limits and reporting limits typically achieved using the analysis method are specified in the QAPP (Appendix A).

### **3.7 DQO Step 7 – Develop the Plan for Obtaining Data**

The basic sampling and analysis approach described in DQO Step 5 will be implemented in accordance with the more detailed plans presented in Section 4.0. The plans developed for data collection are considered resource effective approaches that provide the quantities and quality of data needed to answer the subsurface investigation questions consistent with the analytical approach (Step 5) and performance criteria (Step 6) described above.



## 4.0 SAMPLING AND ANALYSIS PLAN

This section presents the SAP for the proposed multi-media investigation that has been developed to characterize the study area in the upper Animas River Valley (Figure 1-2). Additional areas outside of the study area may be investigated, as directed by Sunnyside. Investigations in these areas would be conducted in accordance with this Work Plan.

An adaptive management approach will be employed for this study and for future studies. As such, the investigation(s) will rely on an iterative methodology that allows for modification to the SAP as warranted by site conditions.

The QAPP is provided in Appendix A. Standard operating procedures (SOPs) that describe surface water sampling procedures and methodologies are provided in Appendix B.

### 4.1 Target Analytes

The TAs for this investigation have been identified for surface water, groundwater, sediment pore water, sediment, and other solid phase media such as soil, mine waste, and mineralized rock. The TAs for surface water and sediment pore water are summarized in Table 4-1. The TAs for groundwater are summarized in Table 4-2. The TAs for the sediment and other solid phase media are summarized in Table 4-3.

#### 4.1.1 Surface Water Sampling

A separate high-flow surface water work plan, which includes the high-flow SAP, has been prepared and was used for the high-flow sampling event conducted in May 2015 (Formation, 2015b). Therefore, the following details the low-flow SAP. The proposed low-flow sampling event locations are listed and described in Table 4-4 and are shown on Figure 4-1. Many of the proposed locations have been historically sampled by the USGS or the ARSG. A brief description of each sample location is presented in Table 4-4. Additional samples may be added to the low-flow SAP, as directed by Sunnyside.

The surface water samples will be analyzed for the full suite of TAs listed in Table 4-1. An unfiltered and a field filtered sample will be collected at each of the sampling locations. Sample filtration methods are described in the surface water sampling SOP (Appendix B).

During the study, the data from USGS gauging station 09538000 will be used to document the stream discharge in the Animas River. Manual field discharge measurements will be performed at selected locations to the extent practical. Field discharge measurement methods are described in the QAPP (Appendix A).

The sample collection methods and sample handling, preservation, and custody procedures are described in the QAPP (Appendix A) and the SOPs are included in Appendix B.

#### **4.1.2 Pore Water Sampling**

Pore water samples will be collected during the low-flow surface water sampling event. Pore water samples will be collected at selected low-flow sampling locations (e.g., those proposed by the EPA in its Upper Animas Mining District 2015 SAP/QAPP [EPA, 2015]) and will be paired with the sediment sample locations described in Section 4.1.4 of this Work Plan. The proposed low-flow pore water sampling locations are listed and described in Table 4-5 and are shown on Figure 4-2. Additional samples may be added to the low-flow pore water SAP, as needed..

The pore water samples will be analyzed for the full suite of TAs listed in Table 4-1. The data will be used for evaluating potential ecological impacts of the existing contaminants in the hyporheic zone of the streambed.

The sample collection methods and sample handling, preservation, and custody procedures are described in the QAPP (Appendix A) and the SOPs are included in Appendix B.

#### **4.1.3 Groundwater Sampling**

Groundwater samples will be collected from selected monitoring wells that will be installed pursuant to the Subsurface Investigation Work Plan (Formation Environmental, 2015a).

The groundwater samples will be analyzed for the full suite of TAs listed in Table 4-2. The sample collection methods and sample handling, preservation, and custody procedures are described in the QAPP (Appendix A) and the SOPs are included in Appendix B.

#### **4.1.4 Sediment Sampling**

Sediment samples will be collected during the low-flow surface water sampling event. Sediment samples will be collected at selected low-flow sampling locations (e.g., those proposed by the EPA in Upper Animas Mining District 2015 SAP/QAPP [EPA, 2015]) and will be paired with the sediment pore water sample locations described in Section 4.1.2 of this Work Plan. The proposed sediment locations are listed and described in Table 4-5 and are shown on Figure 4-2. Additional samples may be added to the sediment SAP, as directed by Sunnyside.

The sediment samples will be analyzed for the full suite of TAs listed in Table 4-3. Data will be used for determination of contaminant concentrations in streambed sediments.

The sample collection methods and sample handling, preservation, and custody procedures are described in the QAPP (Appendix A) and the SOPs are included in Appendix B.

#### **4.1.5 Opportunistic Samples**

In addition to the proposed sample locations describe above, opportunistic surface water samples, groundwater samples, pore water samples, sediment samples and other solid phase media samples (e.g., soils, mine waste, mineralized rock) may also be collected to evaluate TA concentrations.

The opportunistic surface water and pore water samples will be analyzed for the full suite of TAs listed in Table 4-1. The opportunistic groundwater samples will be analyzed for the full suite of TAs listed in Table 4-2. The opportunistic sediment and solid phase media samples will be analyzed for the full suite of TAs listed in Table 4-3.

The sample collection methods and sample handling, preservation, and custody procedures are described in the QAPP (Appendix A) and the SOPs are included in Appendix B.

#### **4.2 Sample Labeling**

Each sample that is collected in the field will be labeled for future identification. Sample labels may be filled out as completely as possible by a member of the sampling team prior to the start of the day's field sampling activities. Samples will be labeled with all necessary information on pre-printed waterproof labels using waterproof ink. At a minimum, each sample label shall contain the following information:

- location identification;
- sample identification number (including codes for site location, sample matrix, and sample type, described in further detail below);
- date and time of sample collection;
- analyses required;
- method of preservation, if used;
- sample matrix;
- sample depth, if applicable.

Each sample shall be assigned a unique sample identification number. These numbers are required for tracking the handling, analysis, and verification or validation status of all samples collected during monitoring. Each sample identification number will identify the sampling location and type of sample. Samples to be collected will include planned soil phase media samples and QC samples.

#### 4.2.1 Surface Water, Pore Water and Sediment

For the planned surface water, pore water and sediment samples, sample identification numbers will be assigned using several codes as follows with the appropriate media type:

Sampling Event - Location – Media Type

SS0815-4023-SW

The first field in the identification number identifies the project location and event month and year. This example includes the project location, “SS” (Sunnyside) and an event month and year of “0815” (August 2015).

The second field in the identification number identifies the location of the sample. In this example, “4023” indicates the sampling location ID. Location identifiers have already been established and are included in Tables 4-4 and 4-5.

The third field identifies the sample matrix type. The matrix type is defined as “SW” to designate the matrix is surface water. The matrix type for pore water will be defined as “PW”. The matrix type for sediment will be “SED”.

Note that additional codes may be added as the project proceeds. The additions will be communicated immediately to the field staff and data management team.

The required QC samples are described in the QAPP (Appendix A). For QC samples, sample identification numbers will be assigned using the same coding described above, but also including information needed by the Formation project team to recognize the field QC samples, for example:

Sampling Event – Location - Matrix

SS0815-8000-SW

[where 8000 is for a non-existent sample location]

For multiple QC samples, the location ID will be numbered sequentially for each sample (e.g., rinsate blank = 8001, duplicate = 8002, etc.)

Field personnel will record the sample identification code with the type of QC sample (e.g., rinsate blank) and the time of sample collection in field log books.

Samples will be immediately labeled in the field and sample numbers shall be recorded at the time of sampling in field notes and on field data collection forms.

#### 4.2.2 Groundwater

For the planned groundwater samples, sample identification numbers will be assigned using several codes as follows with the appropriate media type:

Sampling Event - Location – Media Type

SS0815-MW1-SW

The first field in the identification number identifies the project location and event month and year. This example includes the project location, “SS” (Sunnyside) and an event month and year of “0815” (August 2015).

The second field in the identification number identifies the location of the sample. In this example, “MW1” indicates the sampling location monitoring well ID. Location identifiers have already been established and are included in Tables 4-4 and 4-5.

The third field identifies the sample matrix type. The matrix type is defined as “GW” to designate the matrix is groundwater.

Note that additional codes may be added as the project proceeds. The additions will be communicated immediately to the field staff and data management team.

The required QC samples are described in the QAPP (Appendix A). For QC samples, sample identification numbers will be assigned using the same coding described above, but also including information needed by the Formation project team to recognize the field QC samples, for example:

Sampling Event – Location - Matrix

SS0815-MW100-SW

[where MW100 is for a non-existent sample location]

For multiple QC samples, the location ID will be numbered sequentially for each sample (e.g., rinsate blank = MW100, duplicate = MW101, etc.)

Field personnel will record the sample identification code with the type of QC sample (e.g., rinsate blank) and the time of sample collection in field log books.

Samples will be immediately labeled in the field and sample numbers shall be recorded at the time of sampling in field notes and on field data collection forms.

#### 4.2.3 Other Solid Phase Media

For other planned solid phase media samples, sample identification numbers will be assigned using several codes as follows:

Sampling Event – Location - Depth – Matrix Type

SS0815-01-05-SO

The first field in the identification number identifies the project location and event month and year. This example includes the project location, “SS” (Sunnyside) and an event month and year of “0815” (August 2015).

The second field in the identification number identifies the location of the sample. In this example, “01” indicates the sample location.

The third field identifies the sample depth. The depth is indicated in feet below ground surface (bgs) and in this example “05” indicated 5 feet bgs.

The fourth field identifies the sample matrix type. The matrix type is defined as “SO” to designate the matrix is solid.

Note that additional codes may be added as the project proceeds. The additions will be communicated immediately to the field staff and data management team.

The required QC samples are described in the QAPP (Appendix A). For QC samples, sample identification numbers will be assigned using the same coding described above, but also including information needed by the Formation project team to recognize the field QC samples, for example:

Sampling Event – Location - Depth - Matrix

SS0815-100-01-SO

[where 100 and the depth are for a non-existent sample location]

For multiple QC samples, the depth field will be numbered sequentially for each sample (e.g., rinsate blank = 01, duplicate = 02, etc.)

Field personnel will record the sample identification code with the type of QC sample (e.g., rinsate blank) and the time of sample collection in field log books.

Samples will be immediately labeled in the field and sample numbers shall be recorded at the time of sampling in field notes and on field data collection forms.

#### **4.2.4 Opportunistic**

For opportunistic samples, sample identification numbers will be assigned using the following codes:

Sampling Event - OP - Number

SS0815-OP-01

For opportunistic surface water samples, the first field in the identification number identifies the project location and event month and year. This example includes the project location, "SS" (Sunnyside) and an event month and year of "0815" (August 2015).

The second field in the identification number identifies the sample as an opportunistic sample and the third field identifies the opportunistic sample. Opportunistic samples will be numbered consecutively starting with 1. Field personnel will record the actual sample location and time in field log books.

## 5.0 REPORTING

Upon the completion of the multi-media sampling events, a preliminary report will be prepared to briefly describe the findings of the investigation. These results will be incorporated into an annual report, which will summarize the findings from all phases of investigation conducted during 2015. This report may also include recommendations for additional investigative work.



## 6.0 REFERENCES

EPA, 2002. Guidance for Quality Assurance Project Plans EPA QA/G-5. EPA 240-R-02-009. December.

EPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA QA/G-4. February.

EPA, 2015. Sampling and Analysis Plan/Quality Assurance Project Plan, 2015 Sampling Events, Upper Animas Mining District, San Juan County, Colorado. Final Revision 0, June.

Formation Environmental, 2015a. Subsurface Investigation Work Plan, Mayflower Mill and Tailings Impoundments Area. July.

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Kimball, B.A., Runkel, R.L., and Walton-Day, K., 2010. An Approach to Quantifying Sources, Seasonal Change, and Biogeochemical Processes Affecting Metal Loading in Streams: Facilitating Decisions for Remediation of Mine Drainage. Applied Geochemistry 25 (2010) 728-740.

## Tables

Table 4-1

## Target Analytes for Surface Water and Pore Water Samples

Target Analyte	Total Recoverable (Y/N)	Dissolved (Y/N)	Laboratory Analytical Method	MDL <sup>10</sup> (µg/L)	PQL <sup>10</sup> (µg/L)	Maximum Hold Time (days)	Sample Volume	Preservative
Laboratory Parameters								
Metals								
Aluminum	Y	Y	200.7	30	150	180	250 mL	HNO <sub>3</sub>
Antimony	Y	Y	200.8	0.4	2			
Arsenic	Y	Y	200.8	0.2	1			
Barium	Y	Y	200.8	0.5	2.5			
Beryllium	Y	Y	200.7	10	50			
Cadmium	Y	Y	200.8	0.1	0.5			
Calcium	Y	Y	200.7	100	500			
Chromium	Y	Y	200.7	10	50			
Cobalt	Y	Y	200.8	0.05	0.25			
Copper	Y	Y	200.8	0.5	2.5			
Iron	Y	Y	200.7	20	100			
Lead	Y	Y	200.8	0.1	0.5			
Lithium	Y	Y	200.7	8	40			
Magnesium	Y	Y	200.7	200	1000			
Manganese	Y	Y	200.7	5	25			
Molybdenum	Y	Y	200.8	0.5	2.5			
Nickel	Y	Y	200.8	0.6	3			
Potassium	Y	Y	200.7	200	1000			
Selenium	Y	Y	200.8	0.1	0.5			
Silica	Y	Y	200.7	428	2140			
Silver	Y	Y	200.8	0.05	0.25			
Sodium	Y	Y	200.7	200	1000			
Strontium	Y	Y	200.7	5	25			
Thallium	Y	Y	200.8	0.1	0.5			
Vanadium	Y	Y	200.8	0.2	1			
Zinc	Y	Y	200.7	10	50			
Anions								
Bromide	N	Y	300.0	50	250	28	250 mL	None
Chloride	N	Y	300.0	500	2500			
Sulfate	N	Y	300.0	500	2500			
Flouride	N	Y	300.0	100	500			
Misc.								
Hardness	Y	--	SM2340B	1,500	7,500	180	250 mL	HNO <sub>3</sub>
Alkalinity	Y	--	SM2320B	2,000	10,000	14	500 mL	None
Dissolved Organic Carbon	N	Y	SM5310B	1,000	5,000	28	250 mL	Phosphoric acid, pH < 2
TSS	Y	--	SM2540D	5,000	25,000	7	500 mL	None
TDS	Y	--	SM2540C	10,000	50,000			
Field Parameters								
Temperature	--	--	--	--	--	--	--	--
Turbidity	--	--	--	--	--	--	--	--
pH	--	--	--	--	--	--	--	--
Specific Conductance	--	--	--	--	--	--	--	--
DO	--	--	--	--	--	--	--	--
ORP	--	--	--	--	--	--	--	--

Notes:

1. -- = not applicable

2. Y = Yes

3. N = No

4. DO = dissolved oxygen

5. ORP = oxidation reduction potential

6. TSS = Total suspended sediments

7. TDS = Total dissolved solids

8. MDL = Method detection limit

9. PQL = Practical quantitation limit

10. Targeted MDLs and PQLs are listed. Laboratories routinely adjust these values, and therefore, reported MDLs and PQLs may differ slightly from those listed here

11. mL = milliliter

Table 4-2

## Target Analytes for Groundwater Samples

Target Analyte	Total Recoverable (Y/N)	Dissolved (Y/N)	EPA Method Number	MDL <sup>10</sup> (µg/L)	PQL <sup>10</sup> (µg/L)	Maximum Hold Time (days)	Sample Volume	Preservative
Laboratory Parameters								
Metals								
Aluminum	Y	Y	200.7	30	150	180	250 mL	HNO <sub>3</sub>
Antimony	Y	Y	200.8	0.4	2			
Arsenic	Y	Y	200.8	0.2	1			
Barium	Y	Y	200.8	0.5	2.5			
Beryllium	Y	Y	200.7	10	50			
Cadmium	Y	Y	200.8	0.1	0.5			
Calcium	Y	Y	200.7	100	500			
Chromium	Y	Y	200.7	10	50			
Cobalt	Y	Y	200.8	0.05	0.25			
Copper	Y	Y	200.8	0.5	2.5			
Iron	Y	Y	200.7	20	100			
Lead	Y	Y	200.8	0.1	0.5			
Lithium	Y	Y	200.7	8	40			
Magnesium	Y	Y	200.7	200	1000			
Manganese	Y	Y	200.7	5	25			
Molybdenum	Y	Y	200.8	0.5	2.5			
Nickel	Y	Y	200.8	0.6	3			
Potassium	Y	Y	200.7	200	1000			
Selenium	Y	Y	200.8	0.1	0.5			
Silica	Y	Y	200.7	428	2140			
Silver	Y	Y	200.8	0.05	0.25			
Sodium	Y	Y	200.7	200	1000			
Strontium	Y	Y	200.7	5	25			
Thallium	Y	Y	200.8	0.1	0.5			
Vanadium	Y	Y	200.8	0.2	1			
Zinc	Y	Y	200.7	10	50			
Anions								
Bromide	N	Y	300.0	50	250	28	250 mL	None
Chloride	N	Y	300.0	500	2500			
Sulfate	N	Y	300.0	500	2500			
Fluoride	N	Y	300.0	100	500			
Misc.								
Hardness	Y	--	SM2340B	1,500	7,500	180	250 mL	HNO <sub>3</sub>
Alkalinity	Y	--	SM2320B	2,000	10,000	14	500 mL	None
Dissolved Organic Carbon	N	Y	SM5310B	1,000	5,000	28	250 mL	Phosphoric acid, pH < 2
TSS	Y	--	SM2540D	5,000	25,000	7	500 mL	None
TDS	Y	--	SM2540C	10,000	50,000			
Field Parameters								
Temperature	--	--	--	--	--	--	--	--
Turbidity	--	--	--	--	--	--	--	--
pH	--	--	--	--	--	--	--	--
Specific Conductance	--	--	--	--	--	--	--	--
DO	--	--	--	--	--	--	--	--
ORP	--	--	--	--	--	--	--	--
Ferrous Iron/Total Iron	--	--	Colorimetric Test	--	--	--	--	--

## Notes:

- = not applicable
- Y = Yes
- N = No
- DO = dissolved oxygen
- ORP = oxidation reduction potential
- TSS = Total suspended sediments
- TDS = Total dissolved solids
- MDL = Method detection limit
- PQL = Practical quantitation limit
- Targeted MDLs and PQLs are listed. Laboratories routinely adjust these values, and therefore, reported MDLs and PQLs may differ slightly from those listed here
- mL = milliliter

Table 4-3

## Target Analytes for Solid Phase Samples

Target Analyte	EPA Method Number	MDL <sup>6</sup> (mg/kg)	PQL <sup>6</sup> (mg/kg)	Maximum Hold Time (days)	Sample Volume	Preservative
Laboratory Parameters						
Metals						
Aluminum	6010	3	15	180	Collect 200 grams	None
Antimony	6020	0.2	1			
Arsenic	6020	0.1	0.5			
Barium	6010	0.3	1.5			
Beryllium	6010	1	5			
Cadmium	6010	0.5	2.5			
Calcium	6010	10	50			
Chromium	6020	0.25	1.25			
Cobalt	6010	1	5			
Copper	6010	1	5			
Fluoride (Soluble)	D3761	0.1	0.5	--		
Iron	6010	2	10	180		
Lead	6010	3	15			
Lithium	6010	0.8	4			
Magnesium	6010	20	100			
Manganese	6010	0.5	2.5			
Mercury	7471	0.002-0.005	0.01-0.025	28		
Molybdenum	6010	2	10	180		
Nickel	6010	0.8	4			
Selenium	6020	0.05	0.25			
Silica	6010	42.8	214			
Silver	6010	1	5			
Strontium	6010	0.5	2.5			
Thallium	6020	0.5	2.5			
Vanadium	6020	0.1	0.5			
Zinc	6010	1	5			
Other						
SPLP extraction and analysis for metals listed above	1312/6020 and 7470 <sup>1</sup>	--	--	180	100 grams	None
Acid-Base Accounting (ABA)	600/2-78-054	--	--	--	10 grams	None

## Notes:

1. -- = not applicable
2. SPLP = Synthetic Precipitation Leaching Procedure
3. Leachate solution analyzed for metals/metalloids by EPA Methods 6020 and 7470
4. MDL = Method detection limit
5. PQL = Practical quantitation limit
6. Targeted MDLs and PQLs are listed. Laboratories routinely adjust these values, and therefore, reported MDLs and PQLs may differ slightly from those listed here

Table 4-4

## Low-Flow Surface Water Investigation Sample Locations

Sample Location ID (meters)	Alias			Source Type	Source Name	Concentration Data Source <sup>1</sup>	Zinc (µg/L)	Cadmium (µg/L)	Manganese (µg/L)	Location Description
	ARSG	USGS Database	Kimball et al. (2010)							
CDPHE Water Quality Standard <sup>11</sup>							280	2.2	2,179	
4023	A56	AMIN-4023	--	S	Animas River	ARSG	360	1.80	--	At Animas River Stakeholder Group location A56; right bank access via gravel road
4166	--	AMIN-4166	4166	S	Animas River	Kimball et. al (2010)	490	0.89	988	At Kimball et. al (2010) low-flow and high-flow in-stream sample location 4166; Kimball et. al (2010) Transport site T1; upstream from Arrastra Creek; right bank access via gravel road; limited left bank access
A58-SS	A58	--	--	LBI	Arrastra Creek	ARSG	187.6	1.64	<10	At Animas River Stakeholder Group location A58; in Arrastra Creek upstream of the confluence with the Animas River
4220	A60	--	--	S	Animas River	ARSG	320	1.9	--	At Animas River Stakeholder Group location A60; right bank access via boat ramp immediately downstream of bridge on Colorado Road 52; left bank access by crossing the bridge
4353C	--	--	4353	RBI	Spring	Kimball et. al (2010)	128,000	507	59,400	Stream level spring, At Kimball et. al (2010) right bank inflow sample location 4353; aluminum oxide colored water; right bank access via gravel road
4353A	--	--	--	RBI	Pipe	--	--	--	--	Inflow from pipe on right bank; right bank access via gravel road
4353B	--	--	--	S	Animas River	--	--	--	--	In stream sample; Immediately downstream of pipe inflow and seep inflow; right bank access via gravel road; limited left bank access
4520	--	4520	--	RBI	--	USGS	97,400	385	781,000	At Kimball et al. (2010) low-flow sample location 4520; Marshy ponds with algae near manganocrete
4581	A61	AMIN-4581	--	S	Animas River	ARSG	1,038	2.78	923	At Animas River Stakeholder Group location A61; upstream of Boulder Creek; right bank access via gravel road; limited left bank access
4656	--	AMIN-4656	--	S	Animas River	--	--	--	--	Upstream for Pinicle Gap; right bank access via gravel road; limited left bank access
4734	--	--	--	Mine Adit	Mine Adit	--	--	--	--	At observed mine adit; access via gravel road
4749	--	--	--	S	Animas River	--	--	--	--	Immediately downstream of observed mine adit; right bank access via gravel road; limited left bank access
4916	--	AMIN-4916	4916	S	Animas River	Kimball et. al (2010)	526	1.63	611	At Kimball et. al (2010) low-flow and high-flow in-stream sample location 4916; Kimball et. al (2010) Transport site T2 (low flow)/T3 (high-flow); immediately upstream from the road culverts for Boulder Creek; right bank access via gravel road; limited left bank access
4951	A62	AMIN-4951	4951	RBI	Boulder Creek	ARSG	980	3.9	2,770	At Kimball et. al (2010) low-flow right bank sample location 4951 and Animas River Stakeholder Group location A62; on right bank of the Animas River; at confluence of Boulder Creek and Animas River; immediately downstream of where boulder creek flows across gravel road; access via gravel road
5000	--	--	--	RBI	--	--	--	--	--	At observed right bank inflow; red colored water; access via gravel road
5038	--	--	--	S	--	Kimball et. al (2010)	12,400	13.7	224,000	At Kimball et. al (2010) low-flow sample location 5038; right bank access via gravel road; limited left bank access
5306	A64	AMIN-5306	--	S	Animas River	ARSG	350	1.2	230	At Animas River Stakeholder Group location A64; right bank access via gravel road; limited left bank access
5356	--	--	--	S	--	USGS	17,300	48.6	89,700	At Kimball et al. (2010) low-flow right bank inflow sample location 5356; discharge from slough draining tailings; right bank access via gravel road
5608	--	--	--	S	--	--	--	--	--	At right bank inflow; drainage from Tailings Impoundment 2 area; right bank access via Colorado Road 2
5858	--	AMIN-5858	5858	S	--	Kimball et. al (2010)	4,440	21	21,900	At Kimball et. al (2010) low-flow sample location 5858; small pool near stream; right bank access via Colorado Road 2
5938	A65	--	--	S	Animas River	ARSG	430	1.1	400	At Animas River Stakeholder Group location A65; right bank access via Colorado Road 2; limited left bank access

Table 4-4

Low-Flow Surface Water Investigation Sample Locations

Sample Location ID (meters)	Alias			Source Type	Source Name	Concentration Data Source <sup>1</sup>	Zinc (µg/L)	Cadmium (µg/L)	Manganese (µg/L)	Location Description
	ARSG	USGS Database	Kimball et al. (2010)							
CDPHE Water Quality Standard <sup>11</sup>							280	2.2	2,179	
6013	--	--	--	RBI	Spring	--	--	--	--	Downstream of spring near metal building; access via Colorado Road 2; on private property
6150	--	--	6150	RBI	--	USGS	23,100	89.8	177,000	At USGS low-flow sample location 6150; Red stained right bank discharge
6215	--	--	--	RBI	--	--	--	--	--	Right bank inflow seep; red stained water, right bank access via Colorado Road 2
6274				S	Animas River	--	--	--	--	In Animas river immediately downstream of right bank inflow seep with red stained water; right bank access via Colorado Road 2
6528	--	AMIN-6528	--	S	Animas River	--	--	--	--	Upstream from former Lacawana Bridge; right bank access via Colorado Road 20; limited left bank access
3	--	--	--	RBI	Pond	--	--	--	--	In pond upgradient of reactive treatment wall; right bank access via Colorado Road 20
6768	A66	AMIN-6768	--	S	Animas River	Kimball et. al (2010)	676	2.06	1,400	Near Kimball et. al (2010) low-flow and high-flow in-stream sample location 6745 and Animas River Stakeholder Group location A66; at former Lacawana Bridge; right bank access via Colorado Road 20; limited left bank access
6879				RBI	Wetland					Right bank inflow at wetland near campground
7049	--	--	7049	RBI	--	Kimball et. al (2010)	9,340	45.4	9,570	At Kimball et. al (2010) low-flow sample location 7049; in braid of Animas River during April 29, 2015 recon; right bank access via gravel road; limited left bank access
7688	--	AMIN-7688		S	Animas River	--	--	--	--	At USGS sample location AMIN-7688; right bank access via E 16th Street; limited left bank access
7690	--	--	7750	RBI	--	Kimball et. al (2010)	7,370	39.7	73,300	Approx. 60 meters upstream from Kimball et. al (2010) right bank inflow low-flow sample location 7750; during 4/30/2015 recon event 7750 was part of the main Animas River channel; ditch draining from pond; right bank access via Animas Street; left bank access via CO Road 32
7858	A68	AMIN-7858	7858	S	Animas River	ARGS	1,900	6.2	4,100	At Kimball et. al (2010) low-flow and high-flow sample location 7858 and Animas River Stakeholder Group location A68; Kimball et. al (2010) Transport site T3 (low flow)/T7 (high flow) – at 14th Street bridge with Kimball et. al (2010) stream gauge

Notes:

- 1. USGS = data from United States Geological Survey (Kimball et al., 2010)
- 2. ARSG = data from Animas River Stakeholders Group database (<http://www.animasriverstakeholdersgroup.org/page11.html>)
- 3. Sample results are representative of the date when the maximum historical zinc concentration was reported.
- 4. µg/L = Micrograms per liter
- 5. ft = feet
- 6. **BOLD** Concentration exceeded noted Colorado Water Quality Standard
- 7. S = Stream
- 8. RBI = Right Bank Inflow
- 9. LBI = Left Bank Inflow
- 10. <10 = concentration below noted laboratory reporting limit
- 11. See Table 5-2; the lowest standard reported was selected, not including table value standards

**Table 4-5****Pore Water and Sediment Investigation Sample Locations**

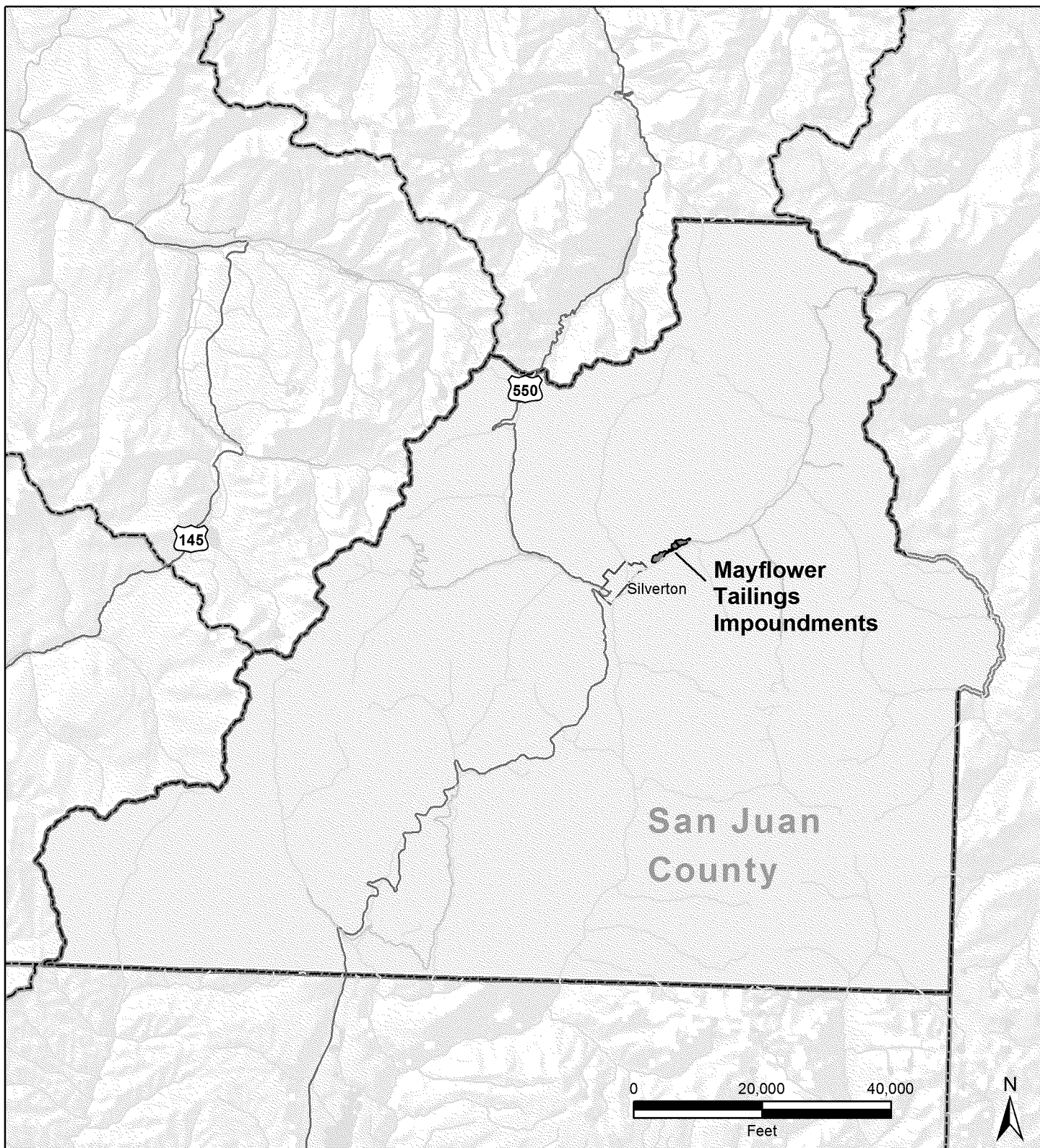
Sample Location ID (meters)	Alias			Source Type	Source Name
	ARSG	USGS Database	Kimball et al. (2010)		
4023	A56	AMIN-4023	--	S	Animas River
A58-SS	A58	--	--	LBI	Arrastra Creek
4220	A60	--	--	S	Animas River
4581	A61	AMIN-4581	--	S	Animas River
5306	A64	AMIN-5306	--	S	Animas River
5938	A65	--	--	S	Animas River
6768	A66	AMIN-6768	--	S	Animas River
7858	A68	AMIN-7858	7858	S	Animas River

Notes:

1. S = Stream
2. LBI = Left Bank Inflow



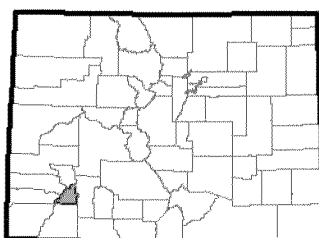
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## Legend

-  Roads
-  Highways
-  Silverton, CO
-  Mayflower Tailings Impoundments
-  Counties

### Location of The Sunnyside Mine in San Juan County, Colorado



## SUNNYSIDE GOLD

SILVERTON, CO

FIGURE 1-1

## SITE LOCATION MAP

DATE: JUL 01, 2015

BY: DKG

FOR: BGH

**FORMATION**  
ENVIRONMENTAL

S:\GIS\5065-001-Sunnyside-UpperAnimas\p1\MultiMedia\SAP\_2015\Fig1\_1\_SiteLocation.mxd





### Legend

- USGS Gauging Station 0938000
- Perennial River or Stream
- Study Area (Approximate Boundary)
- Tailings

Base data sources: 2013 NAIP (National Agriculture Imagery Program) Aerial Photo Rivers and Streams from USGS NHD (National Hydrography Dataset) modified locally using aerial photo

010002000

Feet

N

SUNNYSIDE GOLD

SILVERTON, CO

FIGURE 1-2

SITE PLAN AND STUDY AREA

DATE: JUL 01, 2015

BY: AJ, DKG

FOR: BGH

FORMATION

ENVIRONMENTAL

S:\GIS\5005-001-Sunnyside-UpperAnimas\p\MultiMedia\SAP\_2015\Fig1-2\_SitePlanStudyArea.mxd





### Legend

**Proposed Sample Location**

" Pond

◄ Right Bank Inflow

# In Stream Location

**Rivers and Streams**

— Perennial River or Stream

**Mine Features**

▭ Tailings

Base data sources: 2013 NAIP (National Agriculture Imagery Program) Aerial Photo  
Rivers and Streams from USGS NHD (National Hydrography Dataset)  
modified locally using aerial photo

2013 NAIP (National Agriculture Imagery Program) Aerial Photo

01,0002,000

Feet

SUNNYSIDE GOLD

SILVERTON, CO

FIGURE 4-1

PROPOSED LOW FLOW  
SURFACE WATER SAMPLE  
LOCATIONS

DATE: JUL 01, 2015

BY: DKG

FOR: BGH

FORMATION  
ENVIRONMENTAL

S:\GIS\5005-001-Sunnyside-UpperAnimas\pvt\MultiMedia\SAP\_2015\Fig5-1\_PropLocs\_LowFlow.mxd



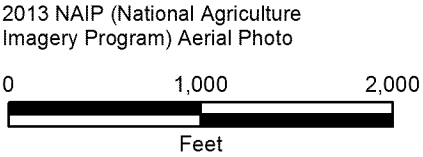


Legend

Sample Location

- In Stream Location
- Perennial River or Stream
- Tailings

Base data sources: 2013 NAIP (National Agriculture Imagery Program) Aerial Photo  
Rivers and Streams from USGS NHD (National Hydrography Dataset)  
modified locally using aerial photo



SUNNYSIDE GOLD

SILVERTON, CO

FIGURE 4-2

PROPOSED PORE WATER  
AND SEDIMENT SAMPLE  
LOCATIONS

DATE: JUL 01, 2015

BY: DKG

FOR: BGH

FORMATION  
ENVIRONMENTAL



## **Appendix A – QAPP**

# **Surface Water, Groundwater, and Solid Phase Media Investigation Work Plan**

## **Mayflower Mill and Tailings Impoundments Area**

### **APPENDIX A - Quality Assurance Project Plan (QAPP)**

**July 2015**

Prepared for:

Sunnyside Gold Corporation  
5075 South Syracuse Street  
Suite 800  
Denver, CO 80237

Prepared by:



2500 55th Street, Suite 200  
Boulder, Colorado 80301

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## LIST OF ACRONYMS

CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CLP	EPA Contract Laboratory Program
COC	Chain of Custody
DO	Dissolved Oxygen
DQOs	Data Quality Objectives
EDD	Electronic Data Deliverable
FISP	Federal Interagency Sedimentation Project
HASP	Health and Safety Plan
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-MS	Inductively Coupled Plasma-Mass Spectrometer
ICV	Initial Calibration Verification
JSA	Job Safety Analysis
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blank
MDL	Method Detection Limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
ND	Not Detected
NFGs	National Functional Guidelines
ORP	Oxidation-Reduction Potential
PQV	Practical Quantitation Verification
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
EPA	United States Environmental Protection Agency

## 1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) comprises Appendix A of the Surface Water, Groundwater, and Solid Phase Media Investigation Work Plan for the Mayflower Mill and Tailings Impoundments Area (Work Plan). The purpose of this QAPP is to describe the quality assurance and quality control (QA/QC) policies and procedures that will be used during data collection and evaluation conducted in support of the surface water, groundwater, and solid phase media investigation proposed for the study area defined in the Work Plan. Additional areas outside of this study area may be investigated, as directed by the Sunnyside Gold Corporation (Sunnyside). Investigations in these areas would be conducted in accordance with this QAPP.

The QAPP describes the measures that shall be employed during the multi-media investigation to assure that data generated are of a known and defensible quality in relation to the overall objectives of the investigation. These measures will assure that the precision and accuracy of program data are known and documented; sample collection, analysis, and reporting are complete; and samples are representative of tested environmental media. This plan also provides guidance for documentation of information collected in the field, including field quality control data; maintenance of documented sample custody and laboratory analytical procedures; and quality control data for data verification and validation.

The QAPP was prepared in accordance with EPA guidance on Quality Assurance Project Plans (EPA, 2002; EPA QA/G-5) and EPA Requirements for Quality Assurance Project Plans (EPA, 2001; EPA QA/R-5). It is comprised of the following four basic project plan elements:

- project management;
- data generation and acquisition;
- data review, validation, and usability; and
- data assessment and oversight.

The subsections that follow provide the four EPA project plan elements (EPA, 2002), and each presents the topics applicable to that element with appropriate Site-specific content, as needed for planning the investigation of environmental media in the study area.

## **2.0 PROJECT MANAGEMENT**

This section addresses project administrative functions and project concerns, goals, and approaches to be followed during implementation of the surface water, groundwater, and solid phase media investigation.

### **2.1 Problem Definition and Background**

The upper Animas River basin and its tributaries are intensely mineralized, and natural weathering of mineralized rock degrades the basin's surface water quality. Streams within the basin that are considered representative of natural-background conditions (i.e., unaffected or minimally affected by mining activity) can be acidic ( $\text{pH} < 3.0$ ) with trace metals concentrations, including zinc, copper, and manganese, above aquatic life standards (USGS, 2007). Environmental conditions in the upper Animas River basin also reflect influences from the extensive historic mining and milling activities that occurred over the past 150 years, including mining in areas upstream of the Mayflower Mill and Tailings Impoundments Area and on the left bank of the Animas River (opposite bank as the Mayflower Mill and Tailings Impoundments area). Mine adits and historic mine waste rock piles are present at numerous locations, and historic mills typically discharged tailings to the Animas and its tributaries.

Previous investigations in the study area have identified elevated levels of metals in the waters of the upper Animas River in the vicinity of the Mayflower Mill and Tailings Impoundments Area. The materials present in the tailings impoundments may be sources of metals to the river, via leaching and subsurface transport by groundwater. However, the current sources of metals and their effects on river water quality remain uncertain. Therefore, additional data are needed to better understand the relationship, if any, between the Mayflower Mill and Tailings Impoundments Area and metals concentrations in surface water of the upper Animas River adjacent to and downstream from this area as well as left bank sources. More specifically, additional physical and chemical data are needed to evaluate the metals concentrations in the surface water of the upper Animas River and in groundwater at and downgradient from this area. Characterization of metals concentrations in nearby Animas River sediments and sediment pore water and soil at selected locations is also needed to better understand the nature and extent of metals contamination within the study area.

### **2.2 Project Description**

The Work Plan presents the rationale and scope of data collection and monitoring activities planned to achieve the investigation objectives described above. The data collection activities associated with the surface water, groundwater, and solid media investigation are described in detail in Section 4.0 of the Work Plan.

## 2.3 Project Organization

The multi-media investigation is being conducted by Sunnyside Gold Corporation (Sunnyside).

Sunnyside's responsibilities include preparation of project planning documents, collection of data needed to complete the surface water, groundwater, and solid media investigation and data analysis and interpretation as needed to complete the investigation.

Sunnyside's project team for the investigation includes:

- Formation Environmental LLC (Formation), Boulder, CO (environmental services contractor); and
- ACZ Laboratories (ACZ), Steamboat Springs, CO (analytical laboratory contractor).

**Sunnyside Program Manager** (Pat Maley, Sunnyside Gold Corporation [Sunnyside]).

Oversees scheduling and management of all technical and non-technical aspects of the project (e.g., field activities, data collection, data analysis, report preparation, scheduling, costing) and serves as primary point of contact with agency representatives.

**Sunnyside Site Manager** (Larry Perino, Sunnyside).

Reports to Sunnyside's Program Manager and serves as the local liaison and provides access and historical knowledge for the mine site.

**Sunnyside Technical Lead** (Linda Schmoll, Ph.D., Sunnyside).

Reports to Sunnyside's Program Manager and reviews all technical aspects of the project, including work plans, QAPPs, data analyses, data reports, etc.

**Sunnyside Field Representative** (Terry Turner, Sunnyside).

Reports to Sunnyside's Program Manager and oversees all field aspects of the project, including sample collection, measurements, and data collection.

**Formation Project Manager** (Brian Hansen, P.E., Formation)

Oversees scheduling and management of all technical and non-technical aspects of the project (e.g., field activities, data collection, data analysis, report preparation, scheduling, costing) and reports to the Sunnyside Program Manager. Directs the Field Investigations Manager and Project QA Manager. Ensures that all field personnel understand the scope of work including QA/QC requirements. Responsible for ensuring that the sampling methods and data analyses

reflected in the Sampling and Analysis Plan (SAP) meet the objectives of the Work Plan. Reviews and approves project plans and all project deliverables.

**Field Investigations Manager** (Nat Beal, P.G., Formation)

Plans and supervises sampling and other field activities and coordinates acquisition of any necessary permits. Schedules and manages various field tasks (e.g., sample collection, measurements, data collection) and is responsible for sample transport to the laboratory. Responsible to the Sunnyside and Formation Project Managers for implementation of field sampling activities, QA/QC measures, and health and safety program requirements defined in the Appendix C of the Work Plan. The Field Investigations Manager is also responsible for ensuring that field staff have appropriate, hands-on training and properly utilize the project Standard Operating Procedures (SOPs; Appendix B of the Work Plan).

**Project QA Manager** (Kathy Tegtmeier, Ph.D., Formation)

Responsible for coordinating the development and approval of the QAPP and its supporting procedures and for maintaining the current, approved version of the QAPP for use on the project. The QA Manager participates in the review and approval of all project deliverables, assists with establishing laboratory contracts, acts as a day-to-day liaison with the laboratories, directs field and laboratory audit activities, coordinates any subsequent corrective and preventive actions, if needed, and communicates regularly with the Formation Project Manager and Field Investigations Manager regarding any laboratory or data validation concerns. The QA Manager will also oversee data validation efforts and coordinate the resolution of any necessary corrective actions resulting from data validation activities, including any quality issues that may be resolved during field activities (i.e., resampling to replace unusable samples).

**ACZ Project Manager** (Max Janicek, ACZ)

Reviews QAPP and ensures laboratory resources are available, reviews final analytical reports produced by the laboratory, coordinates scheduling of laboratory analyses, and supervises in-house chain-of-custody procedures.

## **2.4 Quality Objectives and Criteria for Measurement Data**

This section describes the data needed to address the study objectives as well as the measurement performance criteria established to assess the field and laboratory data quality. Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for precision, accuracy, representativeness, comparability, and completeness (PARCC). The definitions of PARCC are provided below along with the acceptance criteria for data collected in support of this investigation.

### 2.4.1 Data Quality Objectives

The data quality objectives (DQOs) for this program are presented in Section 3.0 of the Work Plan. Consistent with EPA guidelines (EPA, 2006), the DQOs describe the systematic planning of data collection activities to assure that the proper type, quality, and quantity of data are collected. The DQOs will be fulfilled by implementation of these QA and QC activities during data collection in support of the investigation:

- Following specific sampling designs (refer to the Work Plan);
- Adherence to standardized procedures for field measurements, sampling, sample handling, and sample chain of custody (COC) procedures;
- Collection and analyses of field and laboratory QC samples, as discussed in Section 3.4.1 and in Section 3.4.2, respectively;
- Analyses of samples in accordance with standard method protocols selected to meet the project's measurement performance goals (Section 2.4.3) and detectability requirements (Section 3.4.2);
- Adherence to the laboratory analysis methods, and their associated quality control steps, specified for analyses of environmental samples (Section 3.4.2);
- Implementation of laboratory-specific preventative maintenance measures;
- Data review and reduction by the laboratories;
- Data validation; and
- Quality auditing and corrective/preventative action processes, as described in this QAPP.

### 2.4.2 Measurement Performance Criteria - Definitions

The definitions of PARCC are provided below along with the acceptance criteria for data collected in support of the investigation. Equations for calculation of precision, accuracy, and completeness are also provided in Table A2-1.

#### Precision

Precision is the level of agreement among repeated measurements of the same characteristic. There are two general forms of uncertainty. The first is the random error component of the data collection process. The second is inherent stochastic variability, which cannot be eliminated but can be described.

Data precision is assessed by determining the agreement between replicate measurements of the same sample and/or measurements of duplicate samples. The overall random error component of precision is a function of the sampling and analytical precision and is assessed by the analysis of field duplicates. The analytical precision is determined by the analysis of field duplicates by laboratories and by replicate analyses of the same sample. An analytical duplicate is the preferred measure of analytical method precision. When analytes are present in samples



at concentrations below or near the quantitation limit, precision may be evaluated using duplicate analyses of laboratory prepared samples such as duplicate laboratory control samples (LCS/LSCD) and duplicate laboratory matrix spike samples (MS/MSD).

Precision can be measured as relative percent difference (RPD) or as relative standard deviation (RSD; also known as a coefficient of variation). Formulae for both are presented in Table A2-1.

### Accuracy

Accuracy is the degree of difference between the measured or calculated value and the true value. It is a measure of the bias or systematic error of the entire data collection process. Potential sources of systematic errors include:

- sample collection methods;
- physical or chemical instability of the samples;
- interference effects during sample analysis;
- calibration of the measurement system; and
- contamination.

Data accuracy or analytical bias may be evaluated by the analysis of laboratory control samples (LCS) and/or matrix spike (MS) samples, with results expressed as a percentage recovery measured relative to the true (known) concentration (refer to Table A2-1 for percent recovery calculations).

Field equipment and laboratory blanks may be analyzed to assess artifacts introduced during sampling, transport, and/or analysis that may affect the accuracy of the data. In addition, initial and continuing calibration verification samples (ICV and CCV) and initial and continuing calibration blanks (ICB and CCB) may be used to verify that the sample concentrations are accurately measured by the analytical instrument throughout the analytical run.

### Representativeness

Data representativeness is defined as the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or environmental conditions. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. Representativeness of samples shall be achieved through the careful selection of sampling locations and methods. The sampling program described in Section 4.0 of the Work Plan has been designed to provide samples that are representative of the medium being sampled as well as a sufficient number of samples to meet the project DQOs.

### Comparability

Data comparability is defined as the measure of the confidence with which one data set can be compared to another. Comparability is a qualitative parameter but must be considered in the design of the sampling plan and selection of analytical methods, quality control protocols, and data reporting requirements.

### Completeness

Completeness refers to the amount of useable data produced during a sampling and analysis program. The procedures established in this QAPP are designed to ensure, to the extent possible, that data shall be valid and usable. To achieve this objective, every effort shall be made to collect each required sample and to avoid sample loss.

## **2.4.3 Measurement Performance Goals**

This section identifies numerical goals for precision, accuracy, and completeness for the various environmental media. Failure to meet these goals shall be considered in the data validation process described in Section 4.0.

### Precision

Precision shall be determined on field data and laboratory analysis data by the analysis of field duplicates, laboratory replicates, matrix spike and matrix spike duplicate results and evaluation of the RPD for these various paired measurements. The RPD goals for measures of precision associated with the analytical methods are presented in Tables A2-2 through A2-6.

### Accuracy

Sampling accuracy shall be determined by the collection and analysis of equipment blanks, at the frequencies described in Section 3.4.

Laboratory accuracy is determined by the analysis of calibration and method blanks, calibration verification samples, laboratory control samples or standard reference materials, and matrix spike samples. Method blank goals shall be that blanks contain less than the quantitation limit for each target parameter. Accuracy goals for the specific laboratory analysis methods that will be relied on to generate data for the investigation are summarized in Tables A2-2 through A2-6.

### Representativeness

Representativeness is addressed by the description of the sampling techniques and the rationale used to select the sampling locations. Sampling methods are established by the SOPs provided in Appendix B of the Work Plan. Sample representativeness is also evaluated using

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the RPDs for field duplicate results and by a review of the results of field blanks (i.e., equipment blanks as appropriate to sampling methods).

Representativeness of individual sample analyses will be described on the basis of results obtained from associated laboratory quality control samples. The representativeness of sample analyses will be considered acceptable as long as any detectable concentrations of analytes in associated field and method blanks are less than the quantitation limit.

### Comparability

Comparability shall be ensured by analyzing samples obtained in accordance with appropriate SOPs and the referenced standard laboratory analysis methods. All data should be calculated and reported in units consistent with standard reporting procedures so that the results of the analyses can be compared with those of other laboratories, if necessary. In general, data shall be reported in µg/L for water matrices.

### Completeness

The project's completeness goals are 95 percent for analyses of each sample type (i.e., surface water/pore water, groundwater, sediment, and other solid media).

## **2.5 Training Requirements**

Field personnel shall be trained in the requirements of the Work Plan and this QAPP at a project meeting prior to the initiation of field activity. All personnel shall read the Work Plan documents, including this QAPP, prior to the start of field work and shall acknowledge that they have read the documents at the time of the project meeting. In addition, prior to conducting sampling activities, the Field Investigations Manager, or designee, shall review field procedures and sampling requirements in order to better ensure that samples are collected and handled according to Work Plan and QAPP requirements. Field personnel will also be trained in the use of field equipment, decontamination procedures, and COC procedures in accordance with SOPs used for this project (refer to Appendix B of the Work Plan). One hard copy of the current approved version of the Work Plan shall be maintained for ready-reference purposes in the field vehicle or field office. All field team members shall have access to \*.pdf format files of the complete Work Plan through their personal laptop computers.

## **2.6 Documentation and Records**

This section describes the management of project documents and records, including this QAPP. All field documentation will be conducted in accordance with the procedures described in SOP No. 1, *Field Documentation* (Appendix B of the Work Plan).

## 2.6.1 Field Logbooks

Documentation of observations in the field provides information on conditions at the time of sampling and a permanent record of field activities. Field observations and data collected during sampling activities will be recorded with waterproof ink in a permanently bound weatherproof field log book with consecutively numbered pages, or on field forms associated with the individual SOPs found in Appendix B of the Work Plan. Field forms for recording various types of sampling and measurement activities. The appropriate field forms are located in the applicable SOP (refer to Appendix B of the Work Plan). The SOPs also provide instructions for recording field activities at the time of field measurements or sample collection.

Field notebook and data sheet entries will, at a minimum, include the information listed below:

- Project name and number;
- Sample location;
- Data and time of sample collection;
- Sample identification numbers;
- Description of sample (sample matrix or species);
- Number of samples collected;
- Field measurements;
- Field observations and weather conditions;
- Personnel present;
- Sampler's signature; and
- Field filtration activities and equipment, if performed.

In addition, other ancillary information shall be recorded, including:

- personnel and/or other visitors to the sampling site(s);
- weather conditions;
- presence of livestock or wild game; and
- any unusual events.

Changes or deletions in the field book or on the data sheets will be recorded with a single strike mark through the changed entry, with the sampler's initials and the date recording the new entry. All entries must remain legible. Sufficient information should be recorded to allow the sampling event to be reconstructed without having to rely on the sampler's memory.

Completed field forms and logbooks will be copied to the project's quality records (refer to Section 2.6.4) in addition to copies of outgoing COCs and sample shipping documents.

## 2.6.2 Chain of Custody Records

Documentation of sample custody must be maintained. Information on the custody, transfer, handling, and shipping of samples shall be recorded by field personnel on a COC form as specified in SOP No. 2 (Appendix B of the Work Plan), and as described in greater detail in Section 3.2.3 below.

A COC form shall be completed for each set of samples collected daily and shall contain the following information:

- sampler's signature and affiliation;
- program name and identification number;
- date and time of collection;
- sample identification number and matrix;
- analyses requested;
- number of containers;
- signature of persons relinquishing custody, dates, and times;
- signature of persons accepting custody, dates, and times;
- method of shipment; and,
- shipping papers/waybill identification number (as appropriate).

A copy of each as-transmitted COC form shall be retained in the program quality records (refer to Section 2.6.4).

## 2.6.3 Analytical Laboratory Records

Results received from the laboratory will be documented both in report form and in electronic format. Original hard copy and/or electronic reports and data files received from laboratories will be maintained with the program quality records, as described below. Section 4.0 presents the project's laboratory reporting requirements in detail. The final deliverable ("data package" or "report") issued to Sunnyside and Formation will include data necessary to complete validation of laboratory results in accordance with specifications included in Section 4.0.

## 2.6.4 Program Quality Records

Program quality records are defined as completed, legible documents that furnish objective evidence of the quality of items or services, activities affecting quality, or the completeness and quality of data. These records shall be organized and managed by Formation and shall include, at a minimum:

- copies of all bound field logbooks;

- copies of all field documentation forms;
- field copies and original (laboratory) copies of all COC forms;
- incoming and outgoing program correspondence (letters, telephone conversation records, and e-mail messages);
- copies of all laboratory agreements and amendments thereto;
- as-received laboratory data packages (hard copy and/or electronic);
- complete laboratory data validation packages;
- documentation of field and/or laboratory audit findings and any corrective actions;
- draft and final versions of all monthly and quarterly reports; and,
- draft and final delivered versions of the investigation report(s) and supporting procedures such as statistical analyses, numerical models, etc.

The other documentation included in the program's quality records include the approved Work Plan and QAPP, any approved revisions or addendums to the Work Plan and QAPP, and SOPs referred to for field data collection with any updates, revisions, or addendums to those SOPs approved by the Project Managers and Field Investigations Manager to address specific conditions encountered during the field investigation

### **3.0 DATA GENERATION AND ACQUISITION**

The elements in this section address management of data generation and acquisition activities.

#### **3.1 Sampling Design**

The Work Plan provides a detailed description of the sampling design, including the proposed sample locations, and total numbers of samples needed to complete the investigation. Below is a description of the investigation methods. The SOPs included in Appendix B of the Work Plan provide a more detailed description of those procedures, and they also provide information on field documentation and QA activities for the sampling team.

##### **3.1.1 Sampling Locations and Frequencies**

Comprehensive sampling activities, including sampling locations, are summarized in the Work Plan. The number and types of samples that will be collected and sampling locations are detailed in Section 4.0 of the Work Plan.

##### **3.1.2 Surface Water Sampling Methods**

Surface water samples will be collected in accordance with methods specified in SOP No. 5, *Surface Water Sampling* (Appendix B of the Work Plan). One or more of the eight sampling methods described in the SOP will be used for the collection of the surface water samples.

During high-flow conditions, it is anticipated that the majority of the surface water samples will be collected using the “Dipper Method”. The Dipper Method utilizes a sample container attached to a pole that is then dipped into the water body and the sample is collected as a “grab sample” from just below the surface of the water. At the seeps and springs and other features that do not have adequate water for the dipper method the direct method will be used.

During low-flow conditions, it is anticipated that majority of the surface water samples will be collected using the “Dipper Method”. At the seeps and springs and other features that do not have adequate water for the dipper method the direct method will be used. The Equal Discharge Increment Technique (EDITQ) may be used, as needed, and will require protocols specified in the *Operators Manual for the US DH-81 Depth-Integrated Suspended-Sediment Sampler* (FISP, Undated). This technique will be employed during the low-flow event at all locations that warrant its use. It will be at the discretion of the field team lead to determine if the sampling location is suitable to perform this type of sampling using the DH-81. This process of using EDITQ will provide equal sample volumes at each stream vertical sample taken from the cross-section of the stream or river.

An adequate sample volume will be collected from each sample location to meet the required volume for the various laboratory analyses described in Section 3.3 of the QAPP. Depending on the sample collection method used, multiple aliquots may need to be collected to form a composite sample of adequate volume for laboratory analysis. Composite samples will be temporarily contained in an inert disposable container. A portion of the sample collected at each location will be field filtered with a 0.45 micron filter and containerized in the appropriate sample bottles. The non-filtered portion of the sample will then be containerized in the appropriate sample bottles. The remainder of the sample will be used for measurement of field parameters. Field parameters will be measured with a water quality instrument(s) in accordance with the procedures described in SOP No. 5, *Water Quality Sampling* (Appendix B of the Work Plan). Field parameters include: temperature, turbidity, pH, specific conductance, dissolved oxygen (DO), and oxidation reduction potential (ORP). Calibration of the instrument(s) used to measure the field parameters is described in Section 3.6.1 of this QAPP. The water quality meter(s) will be calibrated in accordance with SOP No. 31, *Water Quality Meter Calibration* (Appendix B of the Work Plan).

During sample collection, care will be taken to minimize disturbance of sediment at the bottom of the water body. Samples will be collected in sequential order from the furthest downstream location to the furthest upstream location, unless the sampler does not enter the water, in which case samples can be collected in the order selected by the sampling team(s) based on field logistics. All non-dedicated sampling equipment will be decontaminated between each sample location in accordance with the procedures described SOP No. 7, *Equipment Decontamination* (Appendix B of the Work Plan). Documentation for field sampling is described in Section 2.6 of this QAPP and sample, handling, preservation and custody procedures are described below.

Manual stream discharge measurements will be conducted, where feasible, in accordance with the methods described in SOP No. 6, *Surface Water Discharge Measurement* (Appendix B in the Work Plan). The selection of a discharge measurement method depends on stream flow rate and/or specific channel characteristics.

During high-flow conditions, it may be unsafe for personnel to enter the stream at a particular sample location. The health and safety plan (HASP) for this investigation is included in Appendix C of the Work Plan that includes a job safety analysis (JSA) that describes recommended safe job procedures for stream monitoring. If it is determined, based on the JSA, that conditions are unsafe then a measurement will not be taken.

### 3.1.3 Pore Water Sampling Methods

Pore Water samples will be collected by inserting a PushPoint® sampler into the hyporheic zone and purging until the pore water runs clear in accordance with *Pore Water Sampling*, SOP#FLD-10 (ESAT, 2012a; Appendix B of the Work Plan). The syringe used to extract the pore water will be rinsed three times prior to sample collection with water from the location.



An adequate sample volume will be collected from each sample location to meet the required volume for the various laboratory analyses described in Section 3.3 of the QAPP. Given the limited volume of the syringe, multiple aliquots will be collected to form a composite sample of adequate volume for laboratory analysis. Composite samples will be temporarily contained in an inert disposable container. A portion of the sample collected at each location will be field filtered with a 0.45 micron filter and containerized in the appropriate sample bottles. The non-filtered portion of the sample will then be containerized in the appropriate sample bottles. The remainder of the sample will be used for measurement of field parameters. Field parameters will be measured with a water quality instrument(s) in accordance with the procedures described in SOP No. 5, *Water Quality Sampling* (Appendix B of the Work Plan). Field parameters include: temperature, turbidity, pH, specific conductance, dissolved oxygen (DO), and oxidation reduction potential (ORP). Calibration of the instrument(s) used to measure the field parameters is described in Section 3.6.1 of this QAPP. The water quality meter(s) will be calibrated in accordance with SOP No. 31, *Water Quality Meter Calibration* (Appendix B of the Work Plan).

During sample collection, care will be taken to minimize disturbance of sediment at the bottom of the water body. Samples will be collected in sequential order from the furthest downstream location to the furthest upstream location, unless the sampler does not enter the water, in which case samples can be collected in the order selected by the sampling team(s) based on field logistics. All non-dedicated sampling equipment will be decontaminated between each sample location in accordance with the procedures described SOP No. 7, *Equipment Decontamination* (Appendix B of the Work Plan). Documentation for field sampling is described in Section 2.6 of this QAPP and sample, handling, preservation and custody procedures are described below.

Pore water samples will not be collected during high-flow conditions because of health and safety dangers related to entering fast moving water. Therefore, these samples will only be collected during low-flow conditions. The health and safety plan (HASP) for this investigation is included in Appendix C of the Work Plan and includes a job safety analysis (JSA) that describes recommended safe job procedures for stream monitoring.

#### **3.1.4 Groundwater Sampling Methods**

Groundwater samples and water level measurements will be collected in accordance with methods specified in SOP No. 4, *Groundwater Sampling and Water Level Measurements at Monitoring Wells and Piezometers* (Appendix B of the Work Plan). Field parameters will be measured with a water quality instrument(s) in accordance with the procedures described in SOP No. 4, *Groundwater Sampling and Water Level Measurements at Monitoring Wells and Piezometers* (Appendix B of the Work Plan). Field parameters include: temperature, turbidity, pH, specific conductance, DO, and ORP. Calibration of the instrument(s) used to measure the field parameters is described in Section 3.6.1 of this QAPP. The water quality meter(s) will be calibrated in accordance with SOP No. 31, *Water Quality Meter Calibration* (Appendix B of the Work Plan). All non-dedicated sampling equipment will be decontaminated between each sample location in accordance with the procedures described SOP No. 7, *Equipment*

*Decontamination* (Appendix B of the Work Plan). Documentation for field sampling is described in Section 2.6 of this QAPP and sample, handling, preservation and custody procedures are described below.

### 3.1.5 Sediment Sampling Methods

Sediment samples will be collected using dedicated Teflon scoops and placed in HDPE containers based on the protocols outlined in *Shallow Stream Sediment Sampling* SOP# FLD-06 (ESAT, 2012b; Appendix B of the Work Plan). Several sediment subsamples may be collected to achieve the required sample volume. These samples may be collected from a stretch of creek that is upstream and downstream of the actual sampling location. These subsamples will then be combined and homogenized back at the laboratory. No sampling equipment needs to be decontaminated since the Teflon scoops are not re-used across sampling locations.

Sediment samples will not be collected during high-flow conditions because of health and safety dangers related to entering fast moving water. Therefore, these samples will only be collected during low-flow conditions. The HASP for this investigation is included in Appendix C of the Work Plan and includes a JSA that describes recommended safe job procedures for stream monitoring.

### 3.1.6 Other Solid Phase Media Sampling Methods

Surface or near-surface opportunistic solid phase media samples, including but not limited to soils, mine waste, and mineralized rock, will be collected in accordance with methods specified in the *Standard Operating Procedures for Soil Sampling*, FLD-05 (ESAT, 2012c; Appendix B of the Work Plan). . Samples will be collected using dedicated Teflon scoops. It is expected that no sampling equipment will need to be decontaminated since the Teflon scoops are not re-used across sampling locations. In the event non-dedicated equipment is needed, all non-dedicated sampling equipment will be decontaminated between each sample location in accordance with the procedures described SOP No. 7, *Equipment Decontamination* (Appendix B of the Work Plan).

## 3.2 Sample Handling, Preservation, and Custody

This section describes sample handling requirements and COC procedures from the sample collection step through laboratory analysis and ultimate disposal. Sample custody, packaging, and shipment procedures are described in SOP No. 2, *Sample Custody, Packaging and Shipment* (Appendix B of the Work Plan).

### 3.2.1 Sample Containers, Preservation, and Holding Times

#### Sample Containers

The laboratory will provide new, certified pre-cleaned, prepared sample containers for aqueous sample matrices (i.e., surface water, rinsates, etc.), appropriate to the list of analyses specified on Table A3-1. Solid-phase samples will be contained in sealable, plastic storage bags (e.g., Wirrl Paks or freezer-type Ziplock bags) . After the sample material has been transferred to the plastic bag, the bag will be sealed and labeled and then placed into a second, sealable plastic bag to protect the label.

The mass of material collected and contained for shipping to the laboratory will be sufficient for the list of solid-phase analyses listed in Table A3-1.

#### Sample Preservation and Storage

Samples are preserved in order to prevent or minimize chemical changes that could occur during transit and storage. Sample containers containing appropriate preservative are used to ensure preservation immediately upon sample collection. The contracted laboratories will provide containers and appropriate preservatives (i.e., “pre-preserved” containers), as needed for the analyses to be requested.

Aqueous samples (e.g., surface water, equipment rinsates) submitted for metals/metalloids analyses require preservation upon collection, as specified in Table A3-1. Preservation requirements are associated with the individual analyses to be performed and the referenced analytical methods.

Solid-phase samples will not require chemical preservation, but they will be maintained at low temperature during storage, handling, and shipping to the laboratory. Sample storage requirements are listed in Table A3-1.

#### Sample Holding Times

Sample holding times are established to minimize chemical changes in a sample prior to analysis and/or extraction. A holding time is defined as the allowable time between sample collection and analysis recommended to ensure accuracy and representativeness of analysis results, based on the nature of the analyte of interest and chemical stability factors.

Immediately after collection, samples shall be placed in field coolers with wet ice and/or blue ice. If there is no likelihood that a holding time will be violated, samples may be transferred to a locked refrigerator for one or more days of storage prior to shipping to a laboratory. Transfer to the laboratory for analysis should be prompt to minimize the possibility of exceeding holding times.

Holding times for the chemical constituents for which samples will be analyzed are summarized in Table A3-1. Failure to conduct analyses within the required holding times may result in qualification of associated analytical results and shall prompt appropriate corrective and preventive action measures as outlined in Section 4.4.

### **3.2.2 Sample Handling and Chain of Custody**

#### Sample Handling and Shipping

After collection, sample labels will be completed and the samples will be placed on ice in an insulated cooler. After labeling, each individual sample will be placed in re-closeable freezer-type plastic storage bags. Each sample container will be carefully packaged in a shipping container, typically an ice chest, with Styrofoam® peanuts or other packing material to prevent breakage during shipment. Ice placed in the cooler will be double-bagged to prevent leakage of water. The coolers will be taped shut and the tape will be placed over the custody seal (see below).

#### Chain of Custody

After samples have been collected, they will be maintained under strict COC protocols. The field sampling personnel will complete a COC form (refer to SOP No. 2, Appendix B of the Work Plan) for each shipping container (i.e., cooler, ice chest or other container) of samples to be delivered to the laboratory for analysis. The sampler is responsible for initiating and filling out the COC form. The COC for a shipping container will list only those samples in that shipping container. Information contained on the triplicate, carbonless COC form will include the following:

- Project number;
- Date and time of collection;
- Sample identification number;
- Sample matrix;
- Analyses requested;
- Number of containers/bags for each sample;
- Sample preservation;
- Field filtration, if applicable;
- Sampler's signature and affiliation;
- Signature of persons relinquishing custody, dates, and times;
- Signature of persons accepting custody, dates, and times;
- Method of shipment;

- Shipping air bill number (if the samples are shipped);
- Condition of samples and cooler temperature upon receipt by laboratory; and
- Any additional instructions to the laboratory.

Any documentation, including COCs, placed inside the cooler during sample shipment, should be placed inside a re-closeable plastic bag.

The sampling personnel whose signature appears on the COC is responsible for the custody of the samples from the time of sample collection until custody of the samples is transferred to a designated laboratory, a courier, or to another project employee for the purpose of transporting the sample to the designated laboratory. The sample is considered to be in custody when the sample is: (1) in the direct possession of the sample custodian; (2) in plain view of the sample custodian or (3) is securely locked in a restricted-access area by the sample custodian. Custody is transferred when both parties to the transfer complete the portion of the COC under "Relinquished by" and "Received by." Signatures, printed names, company names, dates and times are required. Upon transfer of custody, the sampling personnel who relinquished the samples will retain the third sheet (pink copy) of the COC. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the COC. Copies, receipts and carbons of Bills of Lading will be retained as part of the permanent documentation in the project file. It is not necessary for courier personnel to sign the COC.

When the analytical laboratory receives the samples, the COC will be immediately signed along with the date and time of receipt. The top sheet (white copy) or a copy of the COC may be returned with the final analytical report. The laboratory will follow appropriate chain-of-custody procedures when shipping any samples to a subcontracted laboratory for analysis. A copy of all inter-lab COCs will be included with the final analytical report.

#### Laboratory Sample Handling and Storage

Upon receipt by the laboratory, the samples will be inspected for sample integrity and proper preservation, including temperature. The COC will be reviewed to verify completeness. Any discrepancies between the COC and sample labels and any problems or questions noted upon sample receipt will be communicated immediately to the Formation QA Manager. The laboratory shall provide the Formation QA Manager with a copy of the COC, and associated sample-receipt information, within 2 working days of receipt of samples. The sample-receipt information routinely provided will include: sample receipt date, sample ids transcribed from the COCs, sample matrix type, list of analyses to be performed for each sample, and verification of sample temperatures and preservation requirements. Broken custody seals, damaged sample containers, sample labeling discrepancies between container labels and the COC form, and analytical request discrepancies shall be noted on the COC form. The Formation QA Manager

shall be notified of any such problems; discrepancies or non-conformances shall be resolved and addressed prior to the samples being released to the laboratory for analysis.

The laboratory will store the samples in a specially designated area, which is clean and maintained at the appropriate preservation temperature, if necessary. The laboratory will be responsible for following their internal custody procedures from the time of sample receipt until sample disposal. At a minimum, the following procedures shall also be in place for laboratory storage of samples:

- samples and extracts shall be stored in a secure area controlled by the laboratory's designated sample custodian;
- samples shall be removed from the shipping container and stored in their original containers unless damaged; damaged samples shall be disposed in an appropriate manner after notifying the Formation QA Manager, and authorization to dispose is received and documented;
- whenever samples are removed from storage, removal shall be documented;
- sample transfers shall be documented on internal COC records;
- samples and extracts shall be stored after completion of analyses in accordance with contractual requirements; and
- Samples shall not be stored with standards or sample extracts.

### **3.3 Analytical Methods**

Samples will be prepared and analyzed using standard laboratory procedures and methods according to performance criteria identified in the following sections.

#### **3.3.1 Sample Preparation**

The laboratory analytical parameters and targeted method detection limits and/or quantitation limits and analytical methods for the laboratory analyses of water samples are specified in Table A3-2. The laboratory analytical parameters and targeted method detection limits and/or quantitation limits and analytical methods for analyses of solid-media samples are specified in Table A3-3.

Preparation of water samples shall be in accordance with the method specifications included in Table A3-1, or method 200.2 for total recoverable metals, as well as standard laboratory practices. Preparation of solid-phase samples, including sediment and soil, shall be in accordance with the method specifications included in Table A3-1.

### 3.3.2 Target Analyses and Methods

The target analytes (TAs) for the project include both laboratory and field parameters and are described in detail in Section 4.1 of the Work Plan. Laboratory parameters for aqueous and solid-phase sample types are listed in Tables A3-2 and A3-3 of this QAPP, respectively.

A copy of the appropriate sample-analysis and method table, by sample type, will be included in each batch of samples submitted to the laboratory for analyses to accurately document the analyses being requested.

### 3.4 Quality Control

There is potential variability in any sample collection, analysis, or measurement activity. This section describes checks that will be performed to evaluate that variability.

#### 3.4.1 Field Quality Control Samples

Field quality control samples are introduced into the measurement process to provide information on transport, storage and field handling biases and on field sampling precision. Field blank samples and field duplicate samples will be collected. Field blank samples may be identified to the laboratory so that they are not used for preparation of an analytical duplicate or matrix spike sample. Descriptions and frequencies of these QC samples are provided below. Table A3-4 summarizes the minimum required frequencies for the field QC samples.

##### Equipment-Rinsate Blanks

Analyses of equipment-rinsate blanks quantify artifacts introduced into the sample during collection. Potential sources of bias or cross-contamination include sampling gloves and sampling equipment that may incidentally come into contact with the sample. An equipment rinsate blank consists of analyte-free, reagent-grade water (e.g., ASTM Type II) poured through the sampling equipment, collected in a clean suite of sample bottles, and preserved as needed.

Equipment rinsates shall be collected whenever sampling equipment is reused (and decontaminated) between sampling locations. In such cases, equipment rinsates will be collected at rate of 1 per 10 field samples (see Table A3-4). The equipment rinsate blanks will be analyzed for total metals analyses as well as hardness, fluoride, chloride, sulfate, bromide and dissolved organic carbon, as listed in Table 3-4. Equipment rinsates collected with solid-phase samples will be analyzed for parameters listed in Table A3-4.

##### Filter Blanks

A filter blank quantifies any artifacts that could be introduced into the sample during filtration. One filter blank will be collected during each separate sampling event at a rate of one filter blank

for each batch of filters used during collection of aqueous samples. The blank sample will consist of one container of filtered water and one container of unfiltered water so that the analysis results can be compared. Analyte-free, reagent-grade water will be poured directly into the sample container for the unfiltered portion, and analyte-free, reagent-grade water will be passed through an unused filter into a separate container for the filtered portion. Both containers will be sent to the laboratory for analyses specified in Table A3-4.

### Field Duplicates

Field duplicates are collected to measure the combined sampling and analytical variability associated with the sample results. Duplicate samples are usually collected simultaneously with or immediately after the corresponding original samples have been collected, depending on the sample type and medium and consistent with detailed instructions in the relevant SOPs for sample collection. In all cases, the same sampling protocol is used to collect the original sample and the field duplicate sample. The field duplicate is analyzed for the same suite of analytical parameters as the original sample.

There are no EPA-recommended criteria for evaluation of field duplicate sample comparability; however, the RPD between the original sample and field duplicate can be calculated for each parameter and compared to the project's precision goal. The TA concentrations reported for the field duplicate pairs will be qualified based on the field duplicate RPD results. Possible causes for the observed variability in duplicate samples should be evaluated and explained in the investigation report.

For aqueous samples, field duplicates will be collected at a rate of 1 per 10 samples. These samples will be analyzed for the TAs listed in Table A3-4. The field-duplicate pairs with RPDs greater than 30% (if sample and duplicate concentrations are  $\geq 5X$  QL) will be qualified as estimated ("J" detects and "UJ" for nondetects) and professional judgment will be used regarding flagging other samples in the data set.

For solid-phase media, field duplicates will be collected at a rate of 1 per 10 samples. These samples will be analyzed for the TAs listed in Table A3-4. For the solid media samples, field-duplicate pairs with RPDs greater than 50% will be qualified as estimated.

### **3.4.2 Laboratory Quality Control Samples**

Laboratory quality control samples are introduced into the measurement process to evaluate laboratory performance and sample measurement bias. Control samples may be prepared from environmental samples or be generated from standard materials in the laboratory. The appropriate type and frequency of laboratory QC samples will be dependent on the sample matrix, analytical method, and the laboratory's SOP. Laboratory QC samples will be analyzed in addition to the calibration samples with each QC batch.



Table A3-5 summarizes the minimum required frequencies for the laboratory QC samples. A laboratory method blank, laboratory control sample, analytical duplicate, and a pair of matrix spike samples should be run in each laboratory QC batch at a frequency of 1 each per 20 field samples shown in Table A3-5. Field staff responsible for collection and shipping of samples to the laboratory shall designate the samples to be used for laboratory QC analyses on the COC forms. In the event that such instructions are not included, the laboratory shall always utilize samples submitted from the investigation for preparation of laboratory duplicates and matrix spike samples used for batch QC analyses.

#### Method Blanks

Method blanks shall be used for the laboratory processes. A method blank is a volume of deionized water that is carried through the entire sample preparation and analysis procedure. The method blank volume or weight shall be approximately equal to the sample volumes or sample weights being processed. Method blanks are used to monitor interference caused by constituents in solvents and reagents and on glassware and other sampling equipment.

Project target analytes must not be detected in laboratory method blanks at concentrations greater than the Quantitation Limit (QL). Method blank contamination, if found, will be addressed in accordance with the response actions given in Tables A2-2 through A2-6, as appropriate to the analytical methods. Method blanks will be evaluated during the data validation process, and associated sample results may be qualified on the basis of blank contamination.

#### Laboratory Control Samples

A laboratory control sample (LCS)/laboratory fortified blank (LFB), or a blank spike, is an aqueous or solid control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the program samples. An LCS/LFB is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix for a stock solution that is different than that used for the calibration standards. The LCS/LFB is the primary indicator of process control used to demonstrate whether the sample preparation and analytical steps are in control, apart from sample matrix effects. LCS/LFB samples will be run with all samples at the frequencies specified herein.

#### Analytical Duplicates

Analytical duplicates are samples that are split at some step in the measurement process and then carried through the remaining steps of the process. Duplicate analyses provide information on the precision of the operations involved.

- Analytical duplicates are a pair of subsamples from a field sample that are taken through the entire preparation and analysis procedure; any difference between the results indicates the precision of the entire method in the given matrix.
- Under certain method protocols (refer to Tables A2-2 through A2-6), the matrix spike is duplicated, to provide a matrix spike duplicate, and serves as the analytical duplicate sample.

Analyses of analytical duplicates and/or matrix spike duplicates monitor the precision of the analytical process.

### Matrix Spikes

A matrix spike is prepared by adding an analyte to a subsample of a field sample before sample preparation and analysis. For multi-analyte methods, a representative suite of the analytes is used in the matrix spike. From the concentrations of the analyte in the spiked and unspiked samples, a percent recovery is calculated. Many samples show matrix effects in which other sample components interfere with the determination of the analyte. The value of the percent recovery indicates the extent of the interference.

Laboratory matrix spike samples are used to evaluate potential sample matrix effects on the accurate quantitation of an analyte using the prescribed analytical method. Percent recoveries of target analytes from matrix spike samples should fall within the prescribed control limits. Matrix interference and other effects may cause low or high percent recoveries in investigative samples; matrix effects may be noted at the same time that recoveries from laboratory control samples indicate acceptable method performance.

Site-specific samples shall be used to prepare the MS/MSD samples. Field sampling personnel will collect extra volume and designate on the COC forms the samples that are to be used for the MS/MSD. Every effort will be made to ensure that these samples are representative of the general sample matrix of samples collected on that sampling data. Equipment rinsates and filter blanks are not designated for MS/MSD.

The laboratories will be instructed to use spike concentrations that are consistent with criteria provided in the National Functional Guidelines for Inorganic Data Validation (EPA, 2004) and any specific instructions provided in the referenced analytical methods.

### Performance Evaluation Samples

Program-specific laboratory performance evaluations via performance evaluation samples are not anticipated as part of this investigation, but may be included later if analytical or validation exercises indicate the presence of potential laboratory QA issues.

### **3.5 Instrument/Equipment Calibration and Maintenance**

In order to ensure continual quality performance of any instruments or equipment, calibration and maintenance shall be performed and recorded as described in this section.

#### **3.5.1 Field Equipment**

Preventative maintenance of field equipment will include routine inspection and either calibration or testing as specified in the relevant SOP or manufacturer's instructions.

All field equipment will be cleaned and safely stored between each use, and any routine maintenance recommended by the equipment manufacturer will also be performed. Equipment will be inspected and the calibration checked (if applicable) before it is transported to a field setting for use. Equipment will be inspected before use and field instruments that fail calibration requirements will be tagged as "nonfunctional" or "defective" and returned to the manufacturer or other supplier for repair or replacement.

#### **3.5.2 Laboratory Equipment**

Instruments used by the laboratory will be maintained in accordance with the laboratory's Quality Assurance Plan and method requirements. All analytical measurement instruments and equipment used by the laboratories shall be controlled by a formal calibration and preventive maintenance program. In addition, each laboratory's preventive maintenance program shall include the following, as a minimum:

- a listing of the instruments and equipment;
- the frequency of maintenance considering manufacturer's recommendations and previous experience with the equipment; and
- a file for each instrument containing a list of spare parts maintained, external contracts, and a listing of the items to be checked or serviced during maintenance.

The laboratory will keep maintenance records and make them available for review, if requested, during laboratory audits. Laboratory preventative maintenance will include routine equipment inspection and calibration at the beginning of each day or each analytical batch, per the laboratory's internal SOPs and method requirements.

#### **Calibration Methods**

Physical and chemical calibrations shall be performed within each laboratory as specified by the EPA Methods, instrument manufacturer's guidelines, and this project's calibration requirements for the requested EPA methods, which are summarized in Tables A2-2 through A2-6. When laboratory measurement instruments do not meet the calibration criteria of the laboratory's Quality Assurance Plan and/or EPA method, then the calibration data will be reviewed using the

NFGs (EPA, 2004) and will be qualified accordingly. Calibration records and demonstration of acceptable calibration results will be required elements of the laboratory's data reporting. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing QC activities. These records will be filed at the location where the work is performed and will be subject to QA audit.

Calibration procedures for a specific laboratory instrument will consist of initial calibration (blank and standards), initial calibration verification (ICV) and continuing calibration verification (CCV). All analyses will be governed by the appropriate laboratory SOPs, and appropriate calibration procedures and frequencies can be found in each SOP.

For a summary of the calibration procedures for individual methods, refer to Tables A2-2 through A2-6. Calibration and quality control sample procedures for trace metals analysis by EPA Method 200.8 (inductively coupled plasma-mass spectrometer [ICPMS]) are provided in Table A2-2, for trace metals analysis by EPA Method 200.7 (inductively coupled plasma [ICP]) in Table A2-3, and for mercury by EPA Method 245.1 (cold-vapor atomic absorption [CVAA]) in Table A2-4. Calibration and quality control sample procedures for alkalinity by Method SM2320B are provided in Table A2-5. Calibration and quality control sample procedures for bromide and sulfate by EPA Method 300.0 are provided in Table A2-6.

Calibration and quality control procedures for trace metals analysis of solid-phase samples, including sediment and soil, are specified in Tables A2-2 through A2-4 of the Subsurface Investigation Work Plan, Appendix A, Quality Assurance Project Plan (Formation 2015).

### **3.6 Acceptance Requirements for Supplies and Consumables**

All supplies and consumables received for a project (e.g., sample bottles, calibration standards) will be checked for damage and other deficiencies that would affect their performance. All inspections should be documented and a copy of the inspection should be kept in the project's file.

### **3.7 Criteria for Use of Existing, Non-Direct Measurement Data**

Previous investigations may provide environmental data that are relevant to this investigation. These data are summarized in the Subsurface Investigation Work Plan (Formation Environmental, 2015) and will be used to the fullest extent practicable in the on-going investigation and considering the data quality.

### **3.8 Data Management**

The program quality records will be maintained by Sunnyside's contractor, Formation, in its Boulder, CO office. These records, either electronic or hard copy in form, shall include:

- Project work plans, including this QAPP, with any approved modifications, updates, and addendums;
- Field documentation (including well logs, GPS data on monitoring locations including surveyed elevations of monitoring wells);
- COC records;
- Laboratory documentation (results received from the laboratory will be documented both in report form and in an electronic format);
- Data validation reports;
- Data Summary Reports; and
- Final project reports/deliverables.

Hard-copy field and laboratory records shall be maintained in the project's central data file, where original field and laboratory documents are filed chronologically for future reference. These records are also scanned to produce electronic copies in \*.pdf format. The electronic versions of these records are maintained on Formation's central server system with backup scheduled on a daily basis.

A key element of the project's data management process is maintenance of an electronic database that is used to store relevant environmental sampling data, including existing data considered usable to support the investigation (i.e., non-direct measurement data), in a consistent, readily retrievable format. Microsoft® Access will be used for the data structure and query support, and a designated Database Manager will ensure the security and integrity of electronically stored data. The project's electronic database will be maintained on a central server system with data backup scheduled on a daily basis.

The project database will serve as a source of data for the data presentation and analysis tasks performed to support the surface water, groundwater, and solid media investigation. The database will incorporate, at a minimum, sample collection information (e.g., sample identification, location, date and time of sample collected, matrix) and laboratory analytical fields specified in the project EDD requirements (Table A3-6).

Prior to incorporation of field and laboratory data into the project database, the data and supporting documentation shall be subject to appropriate review, as described below in Section 4.0, to ensure the accuracy and completeness of original data records. Field data that has been reviewed in a hard-copy format will be entered into electronic data files for upload to the project database. All manual data entry into an electronic format will be reviewed by a separate party before such data are incorporated into the project's database (see Section 4.1). Laboratory EDDs and related data packages will be reviewed as part of the data validation process, as described in Sections 4.2 and 4.4.

Following these review steps, field and laboratory electronic data files will be imported to the project database. The data validator(s) (refer to Section 4.4) will add qualifiers and related information to the EDD file/database, for reference by all data users. The EPA flags, Reason Codes, and final, qualified data will be uploaded from electronic files that the data validators populate and return to Sunnyside/Formation, as discussed in Section 4.0. Standardized data import formats and procedures will be used to upload both field and laboratory data into the electronic database. At this time, standardized station identifiers, parameter names, numerical formats, and units of measure are applied to the original information to facilitate comparability across all datasets and within the database.

## **4.0 DATA REVIEW, VALIDATION AND USABILITY**

The following sections address the final project checks conducted to confirm that the data obtained meet the project objectives and to estimate the effect of any deviations on data usability.

### **4.1 Field Data Review**

Raw field data shall be entered in field notebooks; and/or sample collection record forms. The field records shall be reviewed for completeness by the Field Investigations Manager, or his/her designated Field Supervisor, at the end of each day. The overall quality of the field data from any given sampling round shall be further evaluated during the process of data reduction and reporting.

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Field data review will include verification that QC checks are recorded properly in the field logbooks and/or data sheets and that any necessary and appropriate corrective actions were implemented and recorded. Such data will be written into field logbook and/or data sheets immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, the appropriate Field Supervisor will proof the field logbooks and/or data sheets to determine whether any transcription errors have been made by the field crew. If transcription errors have been made, the appropriate Field Supervisor and field crew will address the errors to provide resolution.

Field measurement data will be entered into electronic files for import to the project's database. Data entries will be made from the reviewed field data sheets or logbooks, and all data entries will be reviewed by a separate party before the electronic file is provided to the database manager. Electronic files of field measurement data will be maintained as part of the project's quality records.

### **4.2 Laboratory Data Review**

Internal, laboratory-data reduction procedures will be according to the laboratory's Quality Management Plan. At a minimum, paper records shall be maintained by the analysts to document sample identification number and the sample tag number with sample results and other details, such as the analytical method used (SOP #), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. These records shall be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these records by the laboratory QA Manager takes place prior to final data reporting to Sunnyside.

QC data (e.g., laboratory duplicates, LCS/LFB, MSs, and MSDs) will be compared to the method and project-specific acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the laboratory QA Officer for review. If approved, data are logged into the project database format. The laboratory shall appropriately flag unacceptable data in the data package.

#### **4.2.1 Laboratory Data Reporting Requirements**

The laboratories shall prepare complete data packages for transmittal of results and associated quality control information to Sunnyside and Formation in general accordance with the following instructions, which are based on the EPA's contract laboratory program (CLP) Statement of Work. Deviations from these specifications may be acceptable provided the report presents all of the requested types of information in an organized, consistent, and readily reviewable format. Laboratories providing data packages for this project shall be responsible for reviewing the following requirements, notifying Formation of any differences between their reports and these requirements, and confirming the acceptability of their intended report content and format with Formation before any laboratory data reports are generated for this project.

Each report will be paginated and organized with a table of contents. A cross reference that correlates the client or field identification as provided on the chain-of-custody document with the laboratory's sample identification will be included.

For each batch of sample results consisting of 20 or fewer samples analyzed together and sharing common QC data, the laboratory data will be presented on a form equivalent to the EPA CLP "Form 1" (see below). Case narratives will be prepared which will include information concerning data that fell outside laboratory acceptance limits, and any other anomalous conditions encountered during sample analysis.

CLP Form 1 contains all required data for field samples. The Form 1 (or equivalent reporting mechanism) will provide the following information:

- Field sample identification;
- Laboratory sample identification;
- Sample result, with appropriate units, MDL, and QL. [analyte concentrations equal to or greater than the method detection limit (MDL) will be reported. Concentrations between the MDL and QL will be flagged as an estimated value ("J") by the laboratory. Parameters that are not detected or not present at concentrations equal to or greater than the MDL are flagged by the laboratory as "U" and interpreted to be not detected at a value equal to or greater than the MDL. Any non-detected value ("U" flagged) will be reported with its QL and MDL.];
- Sample collection and receipt dates;
- Sample preparation date/time;



- Analysis date/time;
- Dilution factor;
- Preparation batch number or identification;
- Analysis batch number or identification;
- Sample matrix and instrument;
- Percent moisture determination; and
- For solid-matrix samples, identify basis of reporting (i.e., wet-weight or dry-weight basis).

The following additional information will be provided with the Form 1s, as applicable for the reported analytical methods. QC batch will be clearly associated with each sample (on the CLP Form specified, or an equivalent reporting mechanism):

- Case narrative;
- Chain-of-custody;
- Summary of all field sample results (Form 1s, or equivalent, as described above);
- Sample results and preparation blank;
- Initial calibration verification (ICV), and continuing calibration verification (CCV);
- Initial calibration blanks (ICB), continuing calibration blank (CCB), and preparation blanks;
- Low-Level Calibration Check Sample Summary, if necessary
- Inductively coupled plasma (ICP) interference check sample or spectral interference check sample (CLP Form IVA-IN);
- Matrix spike (MS) or analytical spike, and when applicable matrix spike duplicate (MSD) or analytical spike duplicate, sample recovery and, when applicable, MS/MSD relative percent difference (RPD);
- Laboratory duplicate precision, where applicable;
- Laboratory control sample (LCS)/laboratory fortified blank (LFB) recovery;
- MDLs;
- ICP interelement correction factors;
- ICP and ICPMS linear ranges;
- Preparation log;
- Analysis run Log;
- ICPMS tunes;
- ICPMS internal standards relative intensity summary;
- Sample log-in sheet; and
- Deliverables inventory sheet.

In addition to this standard data package, the laboratory may be requested to deliver a “Level 4” data package, as detailed below. When requested by the Project Manager, the laboratory’s Level 4 Data Package is to include all items specified above plus instrument raw data and/or documentation of the following additional information:

- Calibration standards (including source, preparation date).
- Blanks (ICB, CCB, and preparation).
- ICV, CCV standards.
- Low-Level Calibration Check Sample or Practical Quantitation Verification Standards.
- Interference check samples.
- LCS/LFB.
- Diluted and undiluted samples.
- Dilution factors.
- Sample volumes.
- Laboratory duplicates.
- Matrix spikes (source, concentration, volume).
- Method of standard addition results.
- Instrument identification.
- Analysis date and time.
- All inorganic methods: full raw data printouts from instruments.
- Full run log for each analysis. and
- ICPMS to include: internal standard recoveries, tune data (atomic mass unit [amu] and peak width), and molecular interference check data.

#### **4.2.2 Laboratory Electronic Data Deliverable**

Each data package, as described above, shall be accompanied by an electronic data deliverable (EDD) prepared by the laboratory. The content and format of laboratory EDDs are specified in Table A3-6. Additional laboratory QC data can be included in the EDD as long as the data fields specified in Table A3-6 are also maintained. The last six fields in the table are to be entered by the data validator.

EDDs will be cross checked against corresponding hard-copy data reports to confirm consistency in results reported in these two separate formats. This cross check will take place as part of the data quality review and validation process described in Section 4.4.

### 4.3 Specific Quality Control Assessment Procedures

The accuracy, precision, completeness, and representativeness of analytical data will be described relative to the project's control limits through a process of field and laboratory data quality review and data validation. Results from these reviews will be documented in routine Data Summary Reports prepared for all data users, and any qualification of the data resulting from that review will also be incorporated into the project's electronic database so that all data users are aware of any uncertainties associated with individual results.

### 4.4 Data Quality Review and Validation

Data validation is the process of verifying that qualitative and quantitative information generated relative to a given sample is complete and accurate. Data validation procedures shall be performed for both field and laboratory operations as described below and in SOP No. 20, *Data Review and Validation* (Appendix B of the Work Plan).

#### 4.4.1 Evaluating Field Data

The results of field quality control sample analyses associated with each laboratory data package will be reviewed to allow for evaluation of equipment blanks and other field QC samples and further indications of the data quality. If a problem is identified through the review of field QC data, all related field samples will be identified, and if possible, corrective actions can be instituted and documented. If data are compromised due to a problem identified via field QC sample review, appropriate data qualifications will be used to identify the data for future data users.

The handling, preservation, and storage of samples collected during the sampling program will be monitored on an on-going basis. The project laboratories will document sample receipt including proper containers and preservation at the time samples are logged into their individual laboratory. The sample receipt records (a required data package deliverable) as well as the COC documentation will also be assessed during data validation. Sample handling, storage or preservation problems identified during data validation will result in appropriate qualification of data.

#### 4.4.2 Evaluating Laboratory Chemistry Data

The purpose of chemistry data-quality review and validation is to verify that the data are of known quality, technically valid, defensible, and usable for their intended purpose. The objectives of the data validation process will be to:

- Verify completeness of data packages and corresponding EDDs;
- Assess compliance to project specific procedures and programs;
- Evaluate system process control through review of control charts (if applicable);

- Verify that no systematic errors exist within the data sets;
- Assess field QC samples to determine if sampling has adversely impacted the reported results and, therefore, usability;
- Assess both method and laboratory performance through tabulation of QC outliers; and
- Provide measures of data quality in terms of precision, accuracy, and completeness so that overall usability can be determined.

Data validation will be performed using the general protocols and processes described in the following documents and in SOP No. 20, *Data Review and Validation* (Appendix B of the Work Plan), as applicable to the method calibration and QC limits specified on Tables A2-2 through 2-6 and to the extent possible when non-CLP methods are used:

- Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (NFG; EPA, 2004); and
- Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (USPEA, 2009).

The data packages will be evaluated and qualified for quantitative QC elements (e.g., spike recoveries, method and field blank contamination, initial and continuing calibration blanks, instrument tunes, interference check samples, duplicate sample %RSD, and instrument stability and performance [e.g., initial and continuing calibration results, instrument tuning and internal standard areas]) using summary forms (described above). This validation procedure is equivalent to a "Stage 2B Validation," as defined in the EPA guidance for labeling externally validated data (EPA, 2009).<sup>1</sup> Specific QC elements that will be reviewed include:

- Presence and completeness of COC and sample receipt documentation;
- Sample Index (correlation of field sample ID to laboratory sample ID);
- Laboratory Case Narrative (method deviations and QC anomalies);
- Analytical holding times;
- Method blank;
- Matrix spike recoveries;
- Matrix spike/matrix spike duplicate RPD values;
- Field duplicate RPD values;
- Laboratory duplicate RPD values;
- Summaries of initial and continuing calibration;
- Summaries of instrument blanks (e.g., initial calibration blank, CCB, if specified in method);

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<sup>1</sup> EPA, 2009. Page 6: "A verification and validation based on completeness and compliance checks of sample receipt conditions and BOTH sample-related and instrument-related QC results..."

- Review of reagent/preparation blanks (inorganics);
- Review of Laboratory Control Standards (LCS);
- Instrument stability and performance (e.g., serial dilution);
- Summaries of internal standards;
- Completeness of laboratory documentation for sample receipt, sample analysis, and sample result reporting;
- Interference check samples (ICP analysis); and
- Serial dilutions (ICP analysis), if any.

Formation will indicate data qualifiers applied to individual results and reasons for application of those qualifiers. Definitions of the data qualifiers that may be applied to individual results as a result of data validation are as follows:

- |    |   |
|----|---|
| U  | The analyte was analyzed for, but was not detected above the level of the reported sample QL.   |
| J  | The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample.   |
| J+ | The result is an estimated quantity, but the result may be biased high.   |
| J- | The result is an estimated quantity, but the result may be biased low.  |
| R  | The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample. |
| UJ | The analyte was analyzed for, but was not detected. The reported QL is approximate and may be inaccurate or imprecise.  |

Formation will add the following data to that EDD upon completion of validation:

Field Header "Validation Qualifier": Populate with validation qualifiers specified above and in template reports.

Field Header "Validation Qual Reason": Populate with a specific reason for qualification if EPA codes are not used.

Field Header "Val Status": Populate with a code to indicate if the data has been validated or not.

Field Header "Val Person": Populate with a code to specify the validation contractor and validator.

Field Header "Val Protocol": Populate with a code to refer to for validation procedures (QAPP or NFG, etc.) used.

Field Header "Val Notes": Populate with additional information that is specific to a sample.

Formation will perform a Manual Validation, as defined in the EPA guidance for labeling externally validated data (EPA, 2009), on the data packages generated by the laboratories.

#### **4.5 Data Usability**

Laboratory packages summarizing the data generated for this investigation will be validated as described above. Once validated, the data will be loaded into a project database managed by Formation. Data usability will be determined by Formation based on the results of data validation and overall comparison to DQOs.

#### **4.6 Measurement Data Analysis and Reporting**

Measurement data will be reported in consistent units for each sample matrix to maintain comparability and facilitate data analyses. Concentrations in liquid samples shall be expressed in terms of weight per unit volume such as milligram per liter (mg/L). The number of significant figures in the field and laboratory data presented in the final report shall be consistent with the limits of uncertainty inherent in the measurement or analytical method.

Statistical analyses and other evaluations may be performed that consider the validated data set. The original detected values for parameters with results below the QL may be used as appropriate to the selected statistical methods. Statistical methods may include published methods found in statistical handbooks, textbooks, and EPA or other agency statistical guidance documents.

## **5.0 ASSESSMENT AND OVERSIGHT**

Assessments of data collection and reporting activities are designed to verify that sampling and analyses are performed in accordance with the procedures established in the Work Plan and QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits. Internal audits will be performed by Sunnyside, Formation, or a contracted laboratory. External audits may be performed by the Lead Agency or supporting agencies. Procedures used to conduct internal and external audits shall be consistent with those described in *EPA Guidance on Technical Audits and Related Assessments* (EPA QA/G-7; EPA, 2000).

Performance and systems audits of field and laboratory data collection and reporting procedures are described in this section. Data assessments, such as data verification and validation, were presented in Section 4.0.

### **5.1 Field Performance and System Audits**

Formation's QA Manager, or designee, may conduct an onsite systems and performance audit of field sampling practices at any time during the field data collection activities. Any non-conformances observed in the audit shall be documented and resolved. Additional systems audits or surveillance may be conducted during the remaining field investigations at the discretion of the Formation Project Manager or Formation QA Manager. One field audit per field season is recommended but not required.

#### **5.1.1 Internal Field Audits**

Internal audits of field activities including sampling and field measurements, will be conducted by the Formation QA Manager, or designee. These audits will verify that procedures established in the Work Plan and QAPP, including referenced SOPs (Appendix B of the Work Plan), are being followed.

The internal field audits (systems and performance audits) will include examination of field measurement and sampling records and field instrument operating records; sample collection, handling, decontamination, and packaging activities; and documentation of sampling activities in compliance with the established procedures for each field activity audited. Follow-up audits may be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the investigation. The results of field audits will be documented. The completed field audit report will be kept on file by the Formation QA Manager. After a field audit is conducted, the results of the audit will be shared by the auditor with the field teams prior to additional sampling to enhance sampling performance where applicable.

Findings of these audits will be summarized in an audit report that is given to the Formation Project Manager, Field Investigations Manager, and appropriate Field Supervisor in charge of the audited activities. The audited party will submit a reply addressing each finding cited in the report, the corrective action (if necessary) to be taken, and a schedule for implementation. The Field Investigations Manager is responsible for ensuring that corrective actions are taken.

### **5.1.2 External Field Audits**

External field audits may be conducted by representatives from the Agencies. External field audits may be conducted at any time during the field operations. These audits may or may not be announced and are at the discretion of the Agencies.

External field audits will be conducted according to the field activity information presented in the field SOPs or in the sampling procedures outlined in the Work Plan. Results of the external field audit may document the need for a change to procedures in the Work Plan and/or QAPP and result in the need for an amendment to the Work Plan and/or QAPP.

## **5.2 Laboratory Performance and Systems Audits**

### **5.2.1 Internal Laboratory Audits**

The internal laboratory audit will be conducted by the QA Officer at each laboratory utilized for the investigation. Audits will be performed in accordance with the laboratory's Quality Management Plan.

The internal laboratory system audits will be conducted on an annual basis while the internal lab performance audits will be conducted on a quarterly basis, or as specified in the laboratory's Quality Management Plan.

The internal laboratory system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc. The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The QA Officer from each laboratory utilized for this investigation will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance.

### **5.2.2 External Laboratory Audits**

An external laboratory audit may be conducted by representatives from the Agencies at any time. An external laboratory audit may be conducted prior to the initiation of the sampling and



analysis activities. These audits may or may not be announced, may be conducted at any time and are at the discretion of the Agencies.

External laboratory audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis. Typically, the external laboratory audit will be conducted in the lab so that the staff may be questioned regarding laboratory procedure. A recently produced sample data package will be compared with their SOP to ensure compliance with applicable standards.

### **5.3 Corrective Actions**

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out-of-QC performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment.

Non-conforming equipment, items, activities, conditions and unusual incidents that could affect data quality and attainment of the project's quality objectives will be identified, controlled and reported in a timely manner. For the purpose of this QAPP, a nonconformance is defined as a malfunction, failure, deficiency, or deviation that renders the quality of an item unacceptable or indeterminate in meeting the project's quality objectives.

Corrective action in the laboratory may occur prior to, during and after initial analyses. If the analytical results from laboratory QC samples fall outside of the measurement performance criteria, the laboratory should initiate corrective actions immediately. If the laboratory cannot correct the situation that caused the nonconformance and an out-of-control situation continues to occur or is expected to occur, then the laboratory will immediately contact the Formation QA Manager and request instructions regarding how to proceed with sample analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the Laboratory Project Manager (or designated QA Officer) to approve the implementation of corrective action. These conditions may include dilution of samples, additional sample extract cleanup, automatic re-injection/re-analysis when certain QC criteria are not met, etc.

Completion of any corrective action should be evidenced by data once again falling within prescribed measurement performance criteria. If an error in laboratory procedures or sample collection and handling procedures cannot be found, the results will be reviewed by the Formation QA Manager and Formation Project Manager to assess whether reanalysis or re-sampling is required.

Any corrective actions taken will be documented in writing by either the Laboratory Project Manager or the Formation QA Manager, as appropriate, and reported to the Formation Project Manager. Corrective action records will be included in the program's quality records.

#### **5.4 Corrective Action during Data Validation and Data Assessment**

The Formation QA Manager may identify the need for corrective action during either the data validation or later data assessment/analysis activities. Potential types of corrective action may include re-sampling by the field team, reanalysis of samples by the laboratory, or re-submission of data packages with corrected clerical errors. The appropriate and feasible corrective actions are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded, etc.). Corrective actions of this type will be documented by the Formation QA Manager.

#### **5.5 Quality Assurance Reports to Management**

The deliverables associated with the tasks identified in the Work Plan will contain QA discussions of data quality information collected during the task is summarized. Those reports will be the responsibility of the Formation Project Manager and QA Manager.

The QA discussions will contain, on a routine basis, the results of field and laboratory audits, information generated on the achievement of specific DQOs and a summary of any corrective actions that were implemented and their immediate results on the project. Detailed references to any QAPP modifications will also be highlighted.

## 6.0 QAPP REFERENCES

- EPA, 2000. Guidance on Technical Audits and Related Assessments, EPA QA/G-7.
- EPA, 2001. Requirements for Quality Assurance Project Plans, EPA QA/R-5.
- EPA, 2002. Guidance for Quality Assurance Project Plans EPA QA/G-5. EPA 240-R-02-009. December.
- EPA, 2004. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. EPA 540-R-04-004. October.
- EPA, 2005. Guidance for Developing Ecological Soil Screening Levels, OSWER Directive 9285.7-55. Revised February 2005. <http://www.epa.gov/ecotox/ecossl/>
- EPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G-4, EPA/240/B-06/001. February.
- EPA, 2009. EPA Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use. EPA 540-R-08-005. January.
- ESAT, 2012a. Pore Water Sampling, SOP#FLD-10. TechLaw Inc. ESAT Region 8. Dated March 30.
- ESAT, 2012b. Shallow Stream Sediment Sampling SOP#FLD-06. TechLaw Inc. ESAT Region 8. Dated March 30.
- ESAT, 2012c. Standard Operating Procedures for Soil Sampling, FLD-05. TechLaw Inc. ESAT Region 8. Dated March 30.
- FISP, Undated. Operator's Manual for the US DH-81 Depth-Integrating Suspended Sediment Sampler. Federal Interagency Sedimentation Project (FISP). [http://water.usgs.gov/fisp/docs/Instructions\\_US\\_DH-81\\_010612.pdf](http://water.usgs.gov/fisp/docs/Instructions_US_DH-81_010612.pdf)
- Formation Environmental LLC (Formation), 2015. Subsurface Investigation Work Plan, Mayflower Mill and Tailings Impoundments, prepared in July.
- MacDonald et al., 2000. Development and Evaluation of Consensus-based Sediment Quality Guidelines for Freshwater Ecosystems, *Environmental Chemistry and Toxicology*, vol. 39, pp 20-31.

**QUALITY ASSURANCE PROJECT PLAN  
TABLES**

Table A2-1

## Precision, Accuracy and Completeness Calculation Equations

Characteristic	Formula	Symbols
<b>Precision</b> (as relative percent difference, RPD)	$RPD = \frac{ x_i - x_j }{\frac{x_i + x_j}{2}} \times 100$	$x_i, x_j$ : replicate values of $x$
<b>Precision</b> (as relative standard deviation, RSD, otherwise known as coefficient of variation)	$RSD = \frac{s}{x} \times 100$	$s$ : sample standard deviation $x$ : sample mean
<b>Accuracy</b> (as percent recovery, R, for samples without a background level of the analyte, such as reference materials, laboratory control samples, and performance evaluation samples)	$R = \frac{x}{t} \times 100$	$x$ : sample value $t$ : true or assumed value
<b>Accuracy</b> (as percent recovery, R, for measurements in which a known amount of analyte, a spike, is added to an environmental sample)	$R = \frac{x_s - x}{t} \times 100$	$x_s$ : value of spiked sample $x$ : value of unspiked sample $t$ : true or assumed value
<b>Completeness</b> (as a percentage, C)	$C = \frac{n}{N} \times 100$	$n$ : number of valid data points produced $N$ : total number of samples taken

Table A2-2

## Summary of Calibration and QC Procedures for EPA Method 200.8 (ICPMS) - Aqueous Samples

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria
Sample preservation and holding time	Not applicable	Sample holding time is 180 days for aqueous samples preserved to pH < 2 with nitric acid.	Laboratory will note sample condition on receipt and notify client if criteria are not met.
AMU Check Tune	Prior to initial calibration solution as specified by lab's standard operating procedure (SOP).	Mass calibration < 0.1 amu from the true value; Stability: RSD < 5% for at least five replicate analyses. Peak resolution < 0.75 AMU at 5% peak height.	Retune instrument then reanalyze tuning solution.
Initial calibration (ICAL) for all target analytes (minimum one standard and a blank)	Daily initial calibration prior to sample analysis.	Calibration blank plus 4 non-zero standards. ICAL must be repeated for each batch unless CCV is used for continuing calibration. If the correlation coefficient for the element of interest is not greater than 0.995, then the instrument must be recalibrated and all of the associated samples for that element must be reanalyzed.	Correct problem then repeat initial calibration.
Initial Calibration Verification (ICV)	Immediately after ICAL, before beginning a sample run and from a second source.	All analytes within $\pm 10\%$ of expected value.	Rerun ICV. If that fails, correct problem and repeat ICAL.
Initial Calibration Blank (ICB)	Immediately after ICV.	< 2.2X the Method Detection Limit (MDL) for dissolved 200.8 analyses or < 3X the MDL of the associated analyte.	Recalibrate if ICB is outside of acceptance criteria. Reanalyze all associated samples that are < 10X the blank and > zero since the last compliant CCB.
Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence.	The analyte concentration within $\pm 10\%$ of expected value	Correct problem then repeat CCV and reanalyze all samples since last successful CCV. If CCV recovery was high, "U" samples can be qualified and passed.
Continuing Calibration Blank (CCB)	Immediately after ICB, after every 10 samples, and at the end of the analytical sequence (after every 20 samples for dissolved analyses)	< 3X MDL	Correct problem then reanalyze calibration blank and previous 10 samples. Reanalyze all associated samples that are < 10X the blank and > zero since the last compliant CCB.
Laboratory Reagent Blank	One per analytical batch of 20 or fewer samples of the same matrix	< 2.2X MDL	Correct problem and reanalyze. Recalibrate if ICB is outside of acceptance criteria.
Laboratory Fortified Blank (LFB)	One LFB per analytical batch of 20 or fewer samples	85-115%	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LFB and all samples in the preparation batch. If the LFB recovery is high, "U" samples may be qualified and passed.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 10 samples per matrix - not to be performed using a field blank.	Percent recovery should be within $\pm 30\%$ and Relative Percent Difference (RPD) should be < 20%. If sample is spiked post digestion, percent recovery should be $\pm 15\%$ . MS/MSD recoveries are not applicable if the sample concentration is >4x the spike concentration.	Qualify associated sample results
Field duplicate sample	1 field duplicate per every 10 samples per matrix	Not applicable	Not applicable
Internal Standards (IS)	Every sample; internal standards as specified by method and lab's SOP.	60% - 125% of intensity in the calibration blank.	Dilute by a factor of two and re-analyze. If IS recoveries still out, report undiluted results.
Concentrations between the MDL and QL	All samples	Not applicable	Qualify to indicate value is between MDL and QL.

Note that specific QC procedures may vary based on the laboratory that performs the analyses.

AMU - Atomic Mass Unit

MDL - Method detection limit

QL - Quantitation Limit. May be referred to as "PQL" - Practical Quantitation Limit or "RL" - Reporting Limit.

ppb - parts per billion

RSD - Relative Standard Deviation

Table A2-3

## Summary of Calibration and QC Procedures for EPA Method 200.7 (ICP) - Aqueous Samples

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria
Sample preservation and holding time	Not applicable	Sample holding time is 180 days for aqueous samples preserved to pH $\leq 2$ with nitric acid.	Laboratory will note sample condition on receipt and notify client if criteria are not met.
Initial calibration (ICAL) for all target analytes (minimum one standard and a blank)	Daily initial calibration prior to sample analysis	Calibration blank plus 1 or more non-zero standards. When 3 or more points are used, the criteria is $r^2 > 0.995$ .	Correct problem then repeat initial calibration.
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for calibration and from a second source)	All analytes within $\pm 5\%$ of expected value. When 3 or more points are used, the criteria is $r^2 > 0.995$ .	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
Initial Calibration Blank (ICB)	After ICV	$< 3X$ Method Detection Limit (MDL)	Correct problem and reanalyze. Sample values $> 10X$ ICB may be accepted and qualified if ICB fails high.
Practical Quantitation Verification (PQV)	Daily, after ICAL and before samples are run.	The analyte concentrations within $\pm 30\%$ of expected value.	Correct problem then reanalyze.
Spectral Interference Check Sample	Immediately after PQV.	Recovery must be within $\pm 20\%$ of expected value.	Correct problem and reanalyze.
Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence (at a mid-calibration range concentration).	The analyte within $\pm 10\%$ of expected value	Correct problem then repeat CCV and reanalyze all samples since last successful CCV. Samples $< MDL$ may be accepted and qualified if CCV fails high.
Continuing Calibration Blank (CCB)	Immediately after CCV and after every 10 samples, and at end of the analytical sequence.	$< 3X$ MDL	Correct problem then reanalyze calibration blank and previous 10 samples. Sample values $> 10X$ CCB or $< MDL$ may be accepted and qualified if CCB fails high.
Laboratory Reagent Blank (LRB) / Method Blank	One per analytical batch of 20 or fewer samples of the same matrix.	$< 2.2X$ MDL	Correct problem and reanalyze. Samples values $> 10X$ LRB or $< MDL$ may be accepted and qualified if LRB fails high.
Laboratory Fortified Blank (LFB)/Laboratory Control Sample (LCS)	One LFB per analytical batch of 20 or fewer samples	85-115%	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LFB and all samples in the preparation batch. If LFB fails high, samples $< MDL$ may be accepted and appropriately qualified.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 10 samples per matrix - field blanks may not be used.	Percent recovery should be within $\pm 30\%$ and Relative Percent Difference (RPD) should be $< 20\%$ . If sample is spiked post digestion, percent recovery should be $\pm 15\%$ . MS/MSD recoveries are not applicable if the sample concentration (used for spiking) is $> 4x$ the spike concentration.	Flag associated sample results. If the $RPD \geq 20\%$ samples must be re-prepped and reanalyzed. Ag must have passing RPD in digested samples.
Field duplicate sample	1 field duplicate per every 10 samples per matrix	Not applicable	Not applicable
Concentrations between the MDL and QL	All samples	Not applicable	Qualify to indicate value is between MDL and QL.

Note that specific QC procedures may vary based on the laboratory that performs the analyses.

MDL - Method detection limit

QL - Contract Required Quantitation Limit. May be referred to as "PQL" - Practical Quantitation Limit or "RL" - Reporting Limit.

**Table A2-4**

**Summary of Calibration and QC Procedures for EPA Method 245.1 (Mercury by CVAA) - Aqueous Samples**

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria
Initial calibration (ICAL) for all target analytes (minimum five standards including a blank)	Daily initial calibration prior to sample analysis	Blank plus 4 or more calibration concentrations, correlation coefficient ( $r$ ) $\geq 0.995$	Correct problem then repeat initial calibration.
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for calibration and from a second source)	All analytes within $\pm 20\%$ of expected value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.
Initial Calibration Blank (ICB)	After ICV	Absolute value $\leq 2x$ MDL for each analyte. If $(2x \text{ MDL}) > QL$ , then use the absolute value $\leq QL$ as the criteria instead.	Correct problem and reanalyze.
CRQL Check Standard (CRI)	Daily, after ICAL, after every 20 samples and at end of each analysis run.	Within $\pm 30\%$ of expected value.	Correct problem then reanalyze.
Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence (at a mid-calibration range concentration)	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat CCV and reanalyze all samples since last successful CCV.
Continuing Calibration Blank (CCB)	Before beginning a sample run, after every 10 samples, and at end of the analytical sequence	Absolute value $\leq 2x$ MDL for each analyte. If $(2x \text{ MDL}) > QL$ , then use the absolute value $\leq QL$ as the criteria instead.	Correct problem then reanalyze calibration blank and previous 10 samples.
Method Blank (or preparation blank)	One per analytical batch	Absolute value $\leq QL$	If absolute value is $>QL$ all sample results (excluding field blanks) must be $\geq 10x$ the blank concentration. Otherwise, all samples associated with the blank and $<10x$ blank concentration must be redigested and reanalyzed.



**Table A2-4**

**Summary of Calibration and QC Procedures for EPA Method 245.1 (Mercury by CVAA) - Aqueous Samples**

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria
LCS	One LCS per analytical batch.	Vendor-specified control limits.	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS/SRM and all samples in the preparation batch.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 20 samples per matrix - field blanks may not be used.	Laboratory-determined control limits (but not wider than 75-125% recovery and RPD < 20). MS/MSD recoveries are not applicable if the sample concentration (used for spiking) is >4x the spike concentration.	Flag associated sample results.
Analytical duplicate sample	One duplicate per every 20 samples per matrix	RPD <20% if sample and duplicate concentrations $\geq 5 \times \text{QL}$ . If sample and/or duplicate concentration <5xQL the control limit will be a difference of $\pm \text{QL}$ .	Flag associated sample results.
Field duplicate sample	1 field duplicate per every 10 samples per matrix	Not applicable	Not applicable
Concentrations between the MDL and QL	All samples	Not applicable	Flag as estimated value ("B" flag)

Note that specific QC procedures may vary based on the laboratory that performs the analyses.

MDL - Method detection limit

QL - Quantitation Limit May be referred to as "PQL" - Practical Quantitation Limit or "RL" - Reporting Limit.

Table A2-5

## Summary of Calibration and QC Procedures for Method SM2320B - Aqueous Samples

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria
Sample preservation and holding time	Not applicable	Sample collected and sent to lab at 0 to 6°C and analyzed within 14 days of collection.	Laboratory will note sample condition on receipt and notify client if criteria are not met.
Practical Quantitation Verification	One per analytical batch	within $\pm 50\%$	Correct problem prior to continuing analysis. Otherwise, recalibrate system.
Prep Blank Water (PBW)/Method Blank	1 per batch of 20 or less samples	< PQL	All samples affected by high method blanks (sample < 10X the highest (PBW) must be qualified accordingly.
Laboratory Control Standard Water (LCSW)	Analyze 1 LCSW at the beginning of the run, one after every 20 samples (or less) and one at the end of the analysis.	90-110% recovery	Correct problem then reanalyze.
Analytical Duplicate	One analytical duplicate per every 10 client samples - field blanks may not be used.	RPD $\leq 20\%$	If RPD above control limit, reanalyze associated samples.
Field duplicate sample	1 field duplicate per every 10 samples per matrix	Not applicable	Not applicable

Note that specific QC procedures may vary based on the laboratory that performs the analyses.

PQL - Practical Quantitation Limit

RPD - Relative percent difference

Table A2-6

## Summary of Calibration and QC Procedures for Method 300.0 (Ion Chromatography) - Aqueous Samples

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria
Sample preservation and holding time	Not applicable	Sample collected and sent to lab unpreserved at 0 to 6°C . Samples analyzed 28 days from collection.	Laboratory will note sample condition on receipt and notify client if criteria are not met.
Calibration Standards	Daily (first batch of the day) or when ICV/CCV fail	$r^2 \geq 0.995$	Reanalyze suspect calibration standard. If criteria still not met, then remake standards and recalibrate the instrument.
Initial Calibration Verification (ICV)	One per analytical batch. Immediately following calibration.	Aqueous: 90-110%	If ICV does not meet criteria, retest one time, if problem persists, recalibrate the instrument.
Continuing Calibration Verification (CCV)	Before beginning a sample run if using a continuing calibration, 1 every 10 client samples and 1 at the end of each batch.	Aqueous: 90-110%	Rerun CCV to see if within limits. If both attempts fail, recalibration or two consecutive passing CCVs are required.
Initial Calibration Blanks (ICB)	Immediately following calibration after ICV.	$< \pm 3X$ (Method Detection Limit) MDL	Rerun ICB to see if it meets criteria. If not, then recalibrate instrument.
Continuing Calibration Blank (CCB)	Before beginning a sample run, after the CCV, if using a continuing calibration, after every 10 client samples, and at end of the analytical sequence	$< \pm 3X$ MDL	Correct problem then reanalyze. Samples $< MDL$ can be accepted and reported with the appropriate qualifier if bracketing CCB(s) fails high.
Laboratory Fortified Blank (LFB)/Laboratory Control Sample (LCS)	One per every 20 or fewer client samples.	90-110%	One re-test OK, then, if not within limits, reanalyze all samples. Recalibrate if necessary.
Analytical Spike (Matrix Spike)	One per every 10 analytical samples	90-110%	If instrument QC for other QC samples is within limits, sample results are qualified. If other instrument QC is out of limits, reanalyze samples.
Analytical Duplicate	One per every 10 analytical samples	RPD $< 20\%$ /for duplicate concentrations $> 10$ times MDL	Reanalyze and if within limits, then no impact. If reanalysis results not within limits, then reanalyze associated samples. If duplicate results $< 10X$ MDL, qualify sample results. If the LFB & associated instrument QC pass, then accept qualify the data.
Field duplicate sample	1 field duplicate per every 20 samples per matrix	Not applicable	Not applicable

Note that specific QC procedures may vary based on the laboratory that performs the analyses.

Table A3-1

## Requirements for Sample Preservation and Preparation Techniques, Sample Volumes, and Holding Times

Sample Type	Parameter	Laboratory Analytical Method	Sample Preparation Method	Preservative <sup>1</sup>	Minimum Sample Volume or Mass	Bottle Type	Maximum Holding Time (Days)
Aqueous	Metals and metalloids and Hardness	EPA 200.7 and 200.8 (ICP and ICP-MS) and SM2340B (hardness) by calculation	Field filtered (dissolved), Hot Plate Digestion 200.2 (200.7 and 200.8)	HNO <sub>3</sub>	250 mL	HDPE	180
			Unfiltered (total); Hot Plate Digestion 200.2 (200.7 and 200.8)	HNO <sub>3</sub>	250 mL	HDPE	180
	Mercury	EPA 245.1	Field filtered (dissolved), preparation in accordance with Method 245.1	HNO <sub>3</sub>	250 mL	HDPE	28
			Unfiltered (total); preparation in accordance with Method 245.1	HNO <sub>3</sub>	100 mL	HDPE	28
	Dissolved Organic Carbon	EPA 415.3	Field filtered (dissolved)	Phosphoric acid, pH < 2	250 mL	Glass	28
	Chloride	EPA 300.0	None	None	250 mL	HDPE	28
	Bromide	EPA 300.0	None				
	Fluoride	EPA 300.0	None				
	Sulfate	EPA 300.0	None				
	Alkalinity <sup>2</sup>	SM2320B	None	None	500 mL	HDPE	14
	TSS	SM2540D	None	None			7
	TDS	SM2540C	None	None			7
Solid	Metals and metalloids	6010 6020	Method 3050B Hot plate/acid digestion	None	Collect 250 grams	Plastic bag or glass jar	180
	Mercury	7471	Preparation per Method 7471				28
	Fluoride (soluble)	ASTM D 3761	Preparation per Method 7471				Not defined

## Notes

- In addition to the preservation listed, all samples shall be placed in a cooler with ice that is maintained at temperatures < 6°C but > 0°C (40 CFR 136) following collection and during shipment to the lab.
- The holding time for alkalinity is 14 days.
- HDPE = High-density polyethylene
- Field filtered samples will be filtered with a 0.45 micron filter.
- HNO<sub>3</sub> = Nitric acid
- mL = milliliter

Table A3-2

**Achievable Laboratory Limits and Regulatory Standards for Surface Water and Groundwater Parameters**

Target Analyte	Laboratory Analytical Method	Laboratory Method Detection Limit (µg/L)	Laboratory Reporting Limit <sup>1</sup> (µg/L)
<b>Metals (total and dissolved)</b>			
Aluminum	200.7	30	150
Antimony	200.8	0.4	2
Arsenic	200.8	0.2	1
Barium	200.8	0.5	2.5
Beryllium	200.7	10	50
Cadmium	200.8	0.1	0.5
Calcium	200.7	100	500
Chromium	200.7	10	50
Cobalt	200.8	0.05	0.25
Copper	200.8	0.5	2.5
Iron	200.7	20	100
Lead	200.8	0.1	0.5
Lithium	200.7	8	40
Magnesium	200.7	200	1000
Manganese	200.7	5	25
Molybdenum	200.8	0.5	2.5
Nickel	200.8	0.6	3
Potassium	200.7	200	1000
Selenium	200.8	0.1	0.5
Silica	200.7	428	2140
Silver	200.8	0.05	0.25
Sodium	200.7	200	1000
Strontium	200.7	5	25
Thallium	200.8	0.1	0.5
Vanadium	200.8	0.2	1
Zinc	200.7	10	50
<b>Anions</b>			
Bromide	300.0	50	250
Chloride	300.0	500	2500
Fluoride	300.0	100	500
Sulfate	300.0	500	2500
<b>Other Parameters</b>			
Alkalinity	SM 2320B	2000	10000
Hardness	SM2340B	1500	7500
TSS <sup>4</sup>	SM2540D	5000	25000
TDS <sup>5</sup>	SM2540C	10000	50000
Dissolved Organic Carbon	SM5310B	1000	5000

## Notes:

1. Laboratory reporting limit (RL) is equivalent to the practical quantitation limit (PQL).
2. Colorado Department of Public Health and Environment (CDPHE), 2014. Standards are stated as dissolved phase (e.g., filtered with a 0.45 micron filter). If there is more than one standard then the lowest standard was used.
3. Hardness is not a Target Analyte for groundwater samples.
4. TSS = Total suspended sediments, for surface water samples only
5. TDS = Total dissolved solids, for surface water samples only

Table A3-3

### Laboratory Methods and Detection Limits for Target Analytes in Solid-Media Samples

Target Analyte	EPA Method Number	Method Detection Limit (MDL) <sup>1</sup> (mg/kg)	Quantitation Limit (QL) <sup>1</sup> (mg/kg)
Aluminum	6010	3	15
Antimony	6020	0.2	1
Arsenic	6020	0.1	0.5
Barium	6010	0.3	1.5
Beryllium	6010	1	5
Cadmium	6010	0.5	2.5
Calcium	6010	10	50
Chromium	6020	0.25	1.25
Cobalt	6010	1	5
Copper	6010	1	5
Iron	6010	2	10
Lead	6010	3	15
Lithium	6010	0.8	4
Magnesium	6010	20	100
Manganese	6010	0.5	2.5
Mercury	7471	0.002-0.005	0.01-0.025
Molybdenum	6010	2	10
Nickel	6010	0.8	4
Selenium	6020	0.05	0.25
Silica	6010	42.8	214
Silver	6010	1	5
Strontium	6010	0.5	2.5
Thallium	6020	0.5	2.5
Vanadium	6020	0.1	0.5
Zinc	6010	1	5
<b>Other Parameters</b>			
Fluoride (soluble)	ASTM D 3761	0.1	0.5
SPLP extraction and analysis for metals listed above	1312/6020 and 7470 <sup>2</sup>	--	--
Acid-Base Accounting (ABA)	600/2-78-054	--	--

Notes:

1. Targeted MDLs and QLs are listed. Laboratories routinely adjust these values, and therefore, reported MDLs and QLs may differ slightly from those listed here.

2. Leachate solution analyzed for metals/metalloids by EPA Methods 6020 and 7470.

**Table A3-4**  
**Required Frequencies for Field QC Samples**

Sample Type	Analytes	Analysis Methods	Field Duplicate	Equipment Rinsate Blank	Identify Sample for Laboratory Use in Preparation of Matrix Spike Samples	Filter Blank
Water	Metals and Metalloids (Table A3-2)	200.7 245.1 200.8 SM2340B by calculation	1 / 10 samples	1/10 samples	1/20 samples Extra volume - 500ml bottle Unfiltered, acid-preserved	1 per sampling event or at least 1 per batch of filters used in the field.
	Chloride, Bromide, Fluoride, Sulfate	300.0	1 / 10 samples	1/10 samples	1/20 samples Extra Volume - 500 mL Unfiltered, unpreserved	NA
	Dissolved Organic Carbon	415.3	1 / 10 samples	1 / 10 samples	Not required	NA
	Alkalinity	SM2320B	1 / 10 samples	Not required	Not required	NA
	Total Dissolved Solids <sup>1</sup>	SM2540C	1 / 10 samples	Not required	Not required	NA
	Total Suspended Solids <sup>1</sup>	SM2540D	1 / 10 samples	Not required	Not required	NA
Solid	Metals and Metalloids (Subsurface Investigation QAPP Table A3-2)	6010 6020 7471	1/10 samples	1/10 samples	1/20 samples	NA

Notes:

1. Analysis required for field quality control samples collected with surface water and pore water samples. Not required for quality control samples collected with groundwater.

**Table A3-5**

**Required Frequencies for Laboratory QC Analyses**

Sample Type	Analytes	Analysis Methods	Method Blank*	Laboratory Fortified Blank/Laboratory Control Sample	Analytical Duplicate	Matrix Spike/Matrix Spike Duplicate Pair
Water	Metals and Metalloids (Table A3-2)	200.7 200.8	1/20	1/20	NA	1 pair/ 20 samples
	Hardness	SM2340B by calc				
	Chloride, Bromide, Sulfate, Fluoride	300.0	1/10 <sup>1</sup>	1/20	1/10	1/10 <sup>2</sup>
	Dissolved Organic Carbon	415.3	1/20	1/20	1/10	NA
	Alkalinity	SM2320B	1/20	NA	1/10	NA
	Total Dissolved Solids	SM2540C	1/20	NA	1/10	NA
	Total Suspended Solids	SM2540D	1/20	NA	1/10	NA
Solid	Metals and Metalloids (Subsurface Investigation QAPP Table A3-2)	6010 6020 7471	1/20	1/20	1/10	1 pair/ 20 samples

NA - Not Applicable

<sup>1</sup> A calibration blank instead of a method blank because the samples aren't prepared but are run directly per the method.

<sup>2</sup> Called an analytical spike sample for this method (which is a spiked site sample) but a spike duplicate of this sample is not run per the method.



Table A3-6

## EDD Specifications for the Laboratory

Lab EDD Fields	Description
COCSampleID	Field sample Identification number
SampleDate	Date sample collected
SampleTime	Time sample collected
PreparationMethod	Preparation method number
AnalyticalMethod	Analytical method number
Matrix	Sampling matrix
TorDAnalysis	Total or dissolved analysis (filtered or unfiltered sample)
Basis	Wet/dry basis for analyte reporting
Analyte	Parameter label
Result	Measured concentration
Units	Units of measure
DetLimit	Detection limit
DetLimitType	Detection limit type (e.g., MDL or IDL)
ReportingLimit	Reporting limit
LabQualifier	Parameter value qualifier
Dilution	Dilution factor
LabName	Lab name
SDGNumber	Lab Sample Delivery Group (SDG) number
LabSampleID	Lab sample identification number
ReceivedDate	Date sample received by laboratory
AnalysisDate	Data sample analyzed by laboratory
QQualifier	EPA qualifier (e.g., H or D)
CAS#	Compound name
Validation Qualifier <sup>1</sup>	Qualifier applied based on data validation (J+, UJ, etc)
Validation Qual Reason <sup>1</sup>	If EPA codes not used, a written explanation of the reason qualified
Val Status <sup>1</sup>	Explains if the data has been validated or not
Val Person <sup>1</sup>	Validation contractor and validator (initials)
Val Protocol <sup>1</sup>	Document validator referred to for validation procedures (QAPP or NFG, etc.)
Val Notes <sup>1</sup>	Additional information pertaining to a result

EDD - Electronic data deliverable

IDL - Instrument detection limit

MDL - Method detection limit

SDG - Sample delivery group

<sup>1</sup> Fields to be added by Validator

# Appendix B – Standard Operating Procedures (SOPs)

## LIST OF SOPS

<u>SOP No.</u>	<u>Title</u>
1	Field Documentation
2	Sample Custody, Packaging and Shipment
4	Groundwater Sampling and Water Level Measurements at Monitoring Wells and Piezometers
5	Surface Water Sampling
6	Surface Water Discharge Measurements
7	Equipment Decontamination
20	Data Review and Validation
31	Water Quality Meter Calibration
FLD#-5	Standard Operating Procedure for Soil Sampling
FLD#-6	Shallow Stream Sampling
FLD#-10	Pore Water Sampling

## **SOP No. 1 Field Documentation**

## **STANDARD OPERATING PROCEDURE No. 1**

### **FIELD DOCUMENTATION**

#### **1.0 SCOPE AND APPLICABILITY**

The following Standard Operating Procedure (SOP) describes the protocol for documenting field monitoring activities. The procedures presented herein are intended to be general in nature and are applicable when referenced by site-specific or project-specific planning documents. Appropriate modifications to the procedures may be made to accommodate project-specific protocols when approved in writing or via email by the Project Manager or detailed in a project work plan, sampling plan, or quality assurance project plan.

The objective of this SOP is to establish a consistent method and format to document daily field activities. The resultant field notes and records are intended to provide sufficient information that can be used to recreate the field activities and the collection of environmental data.

#### **2.0 BASIS FOR METHODOLOGY**

The methods and procedures described in this SOP were developed from these sources:

- ☐ ANSI/ASQC E-4 (1994) American National Standards Institute/American Society for Quality Control Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs.
- ☐ ASTM D 6089 (2010) American Society for Testing and Materials (ASTM) ASTM D 6089 Standard Guide for Documenting a Ground-Water Sampling Event.
- ☐ EPA QA/G6 (2007) U.S. Environmental Protection Agency Guidance for Preparation of Standard Operating Procedures (SOPs). EPA/600/B-07/001. Office of Environmental Information, Washington, DC, April.
- ☐ EPA QA/G5 (2002) U.S. Environmental Protection Agency EPA Guidance for Quality Assurance Project Plans. EPA/240/R-02/009. Office of Environmental Information, Washington, DC, December.

### **3.0 PROCEDURES**

#### **3.1 Daily Field Activities**

The field representative actually performing the environmental monitoring or sampling will record all field activities in the field notebook for each day of field work.

Documentation will include:

- A. Project identification;
- B. Date;
- C. Time on job (beginning and ending time);
- D. Weather conditions;
- E. Activity description;
- F. List of personnel and visitors on site;
- G. Safety equipment used and monitoring performed;
- H. Waste storage inventory (if any);
- I. Chronological record of activities and events;
- J. Comments and variances from project work plan;
- K. Content of telephone conversations;
- L. Calibration parameters; and
- M. Signature of the field representative.

The field representative will document all details that would be necessary to recreate the day's activities and events at a later time. The field notebook will be used to document field activities and information that may not be specified on other field record forms. Other activity-specific documentation requirements to be recorded on field record forms are discussed in the Standard Operating Procedure for each activity.

## **4.0 DOCUMENTATION**

### **4.1 Field Record Forms**

In addition to the field notebook, field personnel will complete specific field record forms (which may be in paper or electronic format) applicable to the field activities being conducted. The procedures for completion of activity-specific field record forms are presented in the applicable Standard Operating Procedures. Additional field record forms and applicable procedures may be created for project-specific activities, as necessary.

### **4.2 Records Management**

All original field forms and copies of field notebooks will be filed with the appropriate project records in the project files. Specific field record forms filled out using an electronic device will be printed and filed with the appropriate project records.

## **5.0 QUALITY ASSURANCE/QUALITY CONTROL**

All completed field forms will be reviewed by the Project Manager or project-designated reviewer. Any necessary corrections will be made in pen with a single-line strike out that is initialed and dated.

## **6.0 REFERENCES**

American National Standards Institute/American Society for Quality Control, 1994. Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs. ANSI/ASQC E-4.

American Society for Testing and Materials (ASTM), 2010. ASTM D 6089-97. Standard Guide for Documenting a Ground-Water Sampling Event. American Society for Testing and Materials available online at <http://www.astm.org/>

U.S. Environmental Protection Agency (USEPA), 2007. EPA QA/G6, Guidance for Preparing Standard Operating Procedures (SOPs). EPA/600/B-07/001. Office of Environmental Information, Washington, DC, April. Available online at <http://www.epa.gov/QUALITY/qs-docs/g6-final.pdf>

U.S. Environmental Protection Agency (USEPA), 2002. EPA QA/G5, EPA Guidance for Quality Assurance Project Plans. EPA/240/R-02/009. Office of Environmental

Information, Washington, DC., December.  
<http://www.epa.gov/QUALITY/qs-docs/q5-final.pdf>

Available online at

## **SOP No. 2 Sample Custody, Packaging and Shipment**



## **STANDARD OPERATING PROCEDURE No. 2**

### **SAMPLE CUSTODY, PACKAGING, AND SHIPMENT**

#### **1.0 SCOPE AND APPLICABILITY**

The following Standard Operating Procedure (SOP) describes the protocol for sample custody and packaging and shipment of samples. The procedures presented herein are intended to be general in nature and are applicable when referenced by site-specific or project-specific planning documents. Appropriate modifications to the procedures may be made when approved in writing or via email by the Project Manager.

This SOP applies to any liquid or solid sample that is being transported by the sampler, a courier, or an overnight delivery service.

#### **2.0 BASIS FOR METHODOLOGY**

The methods and procedures described in this SOP were developed from these sources:

- ☐ 49 CFR 173. Shippers – Shippers – General Requirements for Shipping. United States Code of Federal Regulations available online at <http://www.gpoaccess.gov/cfr/index.html>
- ☐ 49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations available online at <http://www.gpoaccess.gov/cfr/index.html>
- ☐ ASTM D 4220-95 (2000). Standard Practices for Preserving and Transporting Soil Samples, American Society for Testing and Materials available online at <http://www.astm.org/>
- ☐ ASTM D 4840-99 (2010). Standard Guide for Sampling Chain-of-Custody Procedures. American Society for Testing and Materials available online at <http://www.astm.org/>

### **3.0 PROCEDURES**

The objectives of this packaging and shipping SOP are to minimize the potential for sample breakage, leakage, or cross contamination; to provide for preservation at the proper temperature; and to provide a clear record of sample custody from collection to analysis.

#### **3.1 Packaging Materials**

The following is a list of materials that will be needed to facilitate proper sample packaging:

- ☐ Chain-of-Custody (COC)/Request for Analysis (RA) forms;
- ☐ Analyte List;
- ☐ Coolers (insulated ice chests) or other shipping containers as appropriate to sample type;
- ☐ Transparent packaging tape;
- ☐ Duct tape or similar (for sealing cooler drain);
- ☐ Zip-lock type bags (note: this is used as a generic bag type, not a specific brand name);
- ☐ Large garbage bags;
- ☐ Protective wrapping and packaging material;
- ☐ Contained ice (packaged and sealed to prevent leakage when melted) or "Blue Ice"; and
- ☐ Chain-of-Custody seals.

#### **3.2 Sample Custody from Field Collection to Laboratory**

After samples have been collected, they will be maintained under chain-of-custody procedures. These procedures are used to document the transfer of custody of the samples from the field to the designated analytical laboratory. The same chain-of-custody procedures will be used for the transfer of samples from one laboratory to another, if required.

The field sampling personnel will complete a COC/RA form and provide an Analyte List for each separate container of samples to be shipped or delivered to the laboratory for chemical or physical (geotechnical) analysis. Information contained on the form will include:

1. Project identification;
2. Date and time of sampling;
3. Sample identification;
4. Sample matrix type;
5. Sample preservation method(s);
6. Number and types of sample containers;
7. Sample hazards (if any);
8. Requested analysis(es);
9. Method of shipment;
10. Carrier/waybill number (if any);
11. Signature of sampling personnel;
12. Name of Project Manager;
13. Signature, name and company of the person relinquishing and the person receiving the samples when custody is being transferred;
14. Date and time of sample custody transfer;
15. Condition of samples upon receipt by laboratory; and
16. Chain of Custody identification number.

The samples will be carefully packaged into shipping containers/ice chests.

The sampling personnel whose signature appears on the COC/RA form is responsible for the custody of a sample from the time of sample collection until the custody of the sample is transferred to a designated laboratory, a courier, or to another employee for the purpose of transporting a sample to the designated laboratory. A sample is

considered to be in their custody when the custodian: (1) has direct possession of it; (2) has plain view of it; or (3) has securely locked it in a restricted access area.

Custody is transferred when both parties to the transfer complete the portion of the COC/RA form under "Relinquished by" and "Received by." Signatures, printed names, company or organization names, and date and time of custody transfer are required. Upon transfer of custody, the sampling personnel who relinquished the samples will retain a copy of the COC/RA form.

### **3.3 Sample Custody within Laboratory**

The designated laboratory will assume sample custody upon receipt of the samples and COC/RA form. Sample custody within the analytical laboratory will be the responsibility of designated laboratory personnel. The laboratory will document the transfer of sample custody and receipt by the laboratory by signing the correct portion of the COC/RA form. Upon receipt, the laboratory sample custodian will note the condition of the samples, by checking the following items:

1. Agreement of the number, identification and description of samples received by comparison with the information on the COC/RA form; and
2. Condition of samples (any bottle breakage; leakage, cooler temperature, etc.).

If any problems are discovered, the laboratory sample custodian will note this information on the "Laboratory Comments/Condition of Samples" section of the COC/RA form, and will notify the sampling personnel or Project Manager immediately. The Project Manager will decide on the final disposition of the problem samples.

The laboratory will retain a copy of the COC/RA form and return an electronic copy to the originator with the final laboratory report of analytical results. The original of the COC/RA form will be retained as part of the permanent documentation in the project file. A record of the history of the sample within the laboratory containing sample status and storage location information will be maintained in a logbook, or a computer sample tracking system, at the laboratory. The following information will be recorded for every sample access event:

1. Sample identification;
2. Place of storage;
3. Date(s) and time(s) of sample removal and return to storage;
4. Accessor's name and title;
5. Reason for access; and
6. Comments/observations (if any).

The laboratory will provide a copy of the logbook or computer file information pertaining to a sample upon request.

### **3.4 Sample Custody during Inter-Laboratory Transfer**

If samples must be transferred from one laboratory to another, the same sample custody procedures described above will be followed. The designated laboratory person (sample custodian) will complete a COC/RA form and sign as the originator. The laboratory relinquishing the sample custody will retain a copy of the completed form. The laboratory receiving sample custody will sign the form, indicating transfer of custody, retain a copy, and return the original record to the originator with the final laboratory report of analytical results. The COC/RA form will be retained as part of the permanent documentation in the project file.

### **3.5 Packaging and Shipping Procedure**

All sample containers will be properly labeled and all samples will be logged on the COC/RA form in accordance with the procedures explained.

All samples will be packed in the cooler so as to minimize the possibility of breakage, cross-contamination, and leakage. Before placing the sample containers into the cooler, all sample bottle caps will be checked and tightened if necessary. A large garbage bag will be placed as a liner inside the cooler and duct tape (or similar) will be used to seal off any drain openings on the inside and/or outside of the cooler. Bottles made of breakable material (e.g., glass) will also be wrapped in protective material (e.g., bubble wrap, plastic gridding, or foam) prior to placement in the cooler. Each sample set or soil

tube liner (for a California, Shelby Tube or Split-spoon Sampler) will be placed into a zip-lock bag to protect from cross-contamination and to keep the sample labels dry. Sample containers will be placed upright in the cooler. Stacking glass sample bottles directly on top of each other will be avoided.

If required by the method, samples will be preserved to 4°C prior to the analysis. Water ice or “blue ice” will be used to keep the sample temperatures at 4°C. The ice will be placed in two zip-lock bags if the samples are to be transported by someone other than the sampler (e.g., a courier or overnight delivery service). The zip-lock bags of ice will be placed in between, on the bottom, and/or on top of the sample containers so as to maximize the contact between the containers and the bagged ice. If the sampler is transporting the samples to the laboratory shortly after sample collection, the water ice may be poured over and between the sample bottles in the cooler.

If there is any remaining space at the top of the cooler, packing material (e.g., Styrofoam pellets or bubble wrap) will be placed to fill the open space of the cooler. After filling the cooler, the garbage bag will be sealed, a copy of the COC/RA form and Analyte List will be placed in a zip-lock bag and taped to the inside of the cooler lid, the top of the cooler will be closed, and the cooler will be shaken to verify that the contents are secure. Additional packaging material will be added if necessary.

When transport to the laboratory by the sampler is not feasible, sample shipment will occur via courier or overnight express shipping service that guarantees shipment tracking and next morning delivery (e.g., Federal Express Priority Overnight or UPS Next Day Air). The same procedures will be followed to pack and fill the cooler and provide the COC/RA form and Analyte List, as if the sampler were transporting the samples to the laboratory. The cooler will be taped shut with packaging tape. Packaging tape will completely encircle the cooler, and chain-of-custody seals will be signed and placed across the front and side of the container opening.

Copies of all shipment records provided by the courier or overnight delivery service will be retained and maintained in the project file.

#### **4.0 DOCUMENTATION AND RECORDS MANAGEMENT**

Daily Field Records or a field notebook with field notes will be kept describing the packaging procedures and the method of shipment. Copies of all shipping records and chain-of-custody records will be retained in the project file.

#### **5.0 QUALITY ASSURANCE**

The Project Manager or designated reviewer will check and verify that documentation has been completed and filed per this procedure.

#### **6.0 REFERENCES**

49 CFR 173. Shippers – General Requirements for Shipments and Packagings. United States Code of Federal Regulations. Available online at <http://www.gpoaccess.gov/cfr/index.html>

49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations. Available online at <http://www.gpoaccess.gov/cfr/index.html>

ASTM D 4220-95 (2000). Standard Practices for Preserving and Transporting Soil Samples, ASTM International, West Conshohocken, PA, 2000. Available online at <http://www.astm.org/>

ASTM D 4840-99 (2010). Standard Guide for Sampling Chain-of-Custody Procedures, ASTM International, West Conshohocken, PA, 2010. Available online at <http://www.astm.org/>

## **SOP No. 4 Groundwater Sampling and Water Level Measurements at Mentoring Wells and Piezometers**



## STANDARD OPERATING PROCEDURE No.

### 4

## GROUNDWATER SAMPLING AND WATER LEVEL MEASUREMENTS AT MONITORING WELLS AND PIEZOMETERS

### 1.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the protocol to be followed during measurement of water levels and depths of monitoring wells and piezometers, and for water quality sampling from monitoring wells. The procedures presented herein are intended to be general in nature and are applicable when referenced by site-specific or project-specific planning documents. Appropriate modifications to the procedures may be made to accommodate project-specific protocols when approved in writing or via email by the Project Manager.

The objectives of the groundwater sampling procedures are to minimize changes in groundwater chemistry during sample collection and to maximize the probability of obtaining a representative, reproducible groundwater sample.

### 2.0 BASIS FOR METHODOLOGY

The methods and procedures described in this SOP were developed from these sources:

- ☐ USEPA Standard Operating Procedure for the Standard/Well-Volume Method for Collecting a Ground-Water Sample from Monitoring Wells for Site Characterization.
- ☐ USEPA Region 1 (2010) Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells. EQASOP-GW-001. Region 1 Low-Stress (Low-Flow) SOP, Revision Number 3, July 30, 1996, Revised January 19, 2010.
- ☐ USEPA (2002) Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers, Ground Water Forum Issue Paper. U.S. Environmental Protection Agency, Technology Innovative Office, Office of Solid Waste and Emergency Response, Washington D.C., EPA 542-S-02-001.
- ☐ U.S. Geological Survey (USGS) (variously dated) National Field Manual

for the Collection of Water-Quality Data: U.S. Geological Survey  
Techniques of Water-Resources Investigations, Book 9, Handbooks for  
Water-Resources Investigations, Chapters A1-A9.

### **3.0 WATER LEVEL AND WELL DEPTH MEASUREMENT PROCEDURES**

Prior to performing water level and well depth measurements, the construction details and previous measurements for each well or piezometer shall be reviewed by the field geologist or other field personnel so any anomalous measurements may be identified. Well construction details and previous measurements shall be available in the field for review.

In general, water-level measurements shall be performed before groundwater is removed from the well by purging or sampling.

#### **3.1 Equipment**

Equipment that may be necessary to perform measurements (depending on measurements to be performed):

- ☐ Well/piezometer construction details;
- ☐ An electronic water-level meter with accuracy of 0.01 foot;
- ☐ Water Level Monitoring Record Sheet, Groundwater Sampling Record or field notebook; and
- ☐ Weighted surveyor's rope (measured to the nearest 0.1 foot).

#### **3.2 Measuring Point**

A measuring point (MP) shall be selected and marked for each monitoring well and piezometer in which water level measurements will be made. Generally, the MP will be on the north side of the top of the well casing. The MP will be permanently marked using an indelible marker or a notch cut into the PVC casing. When the top-of-casing elevation of a monitoring well or piezometer is surveyed, the licensed surveyor shall measure the MP elevation and reference this measurement to an appropriate datum (such as feet above mean sea level).

### 3.3 Water Level Measurements

When water levels are measured to describe the groundwater potentiometric surface, the water level will be measured prior to purging. All wells to be gauged during a monitoring event and used to construct the potentiometric surface should have water levels measured within the same 24-hour period, as practical. All water level measurements will be recorded to the nearest 0.01 foot. Instruments used for each measurement will be noted on the Groundwater Sampling Record (a attached form or similar). Water levels are measured using the electronic probe method, as discussed below.

An electronic probe consists of a contact electrode attached to the end of an insulated electrical cable, and a reel which houses an ammeter, a buzzer, or other closed circuit indicator. The indicator shows a closed circuit and flow of current when the electrode touches the water surface.

The procedure for measuring water levels with an electric probe is as follows:

1. Switch on and test that the battery is charged and set the sensitivity dial to the middle position.
2. Lower the probe into the well until the ammeter or buzzer indicates a closed circuit. Raise and lower the probe slightly until the shortest length of cable that gives the maximum response on the indicator is found.
3. With the cable in this fixed position, note the depth to water from the Measuring Point (MP).
4. Repeat as necessary until at least two identical duplicate measurements are obtained.

Calibration of the electronic probe will be checked at regular intervals as part of regular maintenance measuring the position of the electrode to check that the calibration marks on the electronic probe correspond to those on a weighted surveyor's rope or other suitable measuring device.

### 3.4 Well Depth Measurements

The total depth of a well shall be measured by sounding with a weighted surveyor's rope or other suitable measuring device. For shallow wells, the electronic water-level probe may also be used as a measuring device. Procedures to be followed are specified below.

- A. For calibration, measure the distance between the zero mark on the end of the measuring tape and the bottom of the weight to the nearest 0.1 foot at the beginning of each well depth measurement activity day, and whenever the apparatus is altered.
- B. To measure well depth, lower a weighted tape into the well until the tape becomes slack or there is a noticeable decrease in weight, which indicates the bottom of the well. Care should be taken to lower the tape slowly to avoid damage to the bottom of the well by the weight. Raise the tape slowly until it just becomes taut, and with the tape in this fixed position, note the tape reading opposite the MP to the nearest 0.1 foot. Add the values from the distance from the end of the tape to the end of the weight together, round this number to nearest 0.1 foot, and record the resulting value as "Total Depth (feet [ft], below measuring point [BMP])" on the Groundwater Sampling Record or field notebook.

### 3.5 Documentation and Records Management

Water levels observed in wells selected for the groundwater level monitoring program will be tabulated on the Groundwater Sampling Record form during each monitoring period (in print or electronic format – see attached form) or in the field notebook. The date and time of each measurement will also be recorded in the field. All water level measurements shall be recorded to the nearest 0.01 foot, and all depth measurements shall be noted to the nearest 0.1 foot.

Water level data will be recorded as feet BMP so that water level elevations may be calculated from the depth-to-water measurement (from the MP) and the surveyed elevation of the MP at each well or piezometer. The MP will also be described and documented in the Groundwater Sampling Record and/or field notebook (i.e., top of PVC casing, top of protective casing, or below ground surface).

Well depth measurements may also be recorded on the Groundwater Sampling Record.

## 4.0 GROUNDWATER SAMPLE COLLECTION PROCEDURES

### 4.1 Low Flow Sample Collection

For wells that are sampled for regulatory compliance, a low flow sample collection technique shall be employed whenever possible to ensure that representative groundwater samples are collected from each well. Additionally, low flow sampling is to be the preferred method for groundwater sampling unless site specific conditions warrant a volume-based approach or a non-purge approach such as a HydraSleeve™ (as discussed in Sections 4.2 and 4.3, respectively).

- A. Measure the depth to water (water level must be measured to nearest 0.01 feet) relative to a reference MP on the well casing with an electronic water level indicator or steel tape and recorded.
- B. For wells with non-dedicated equipment (i.e., no dedicated tubing, pump, or docking station), place the pump at the wellhead and slowly lower the pump and tubing down into the well until the location of the pump intake is set to the midpoint of the screened interval, unless otherwise specified in the monitoring plan. Care should be taken to minimize disturbance to the water column during insertion of the pump. A variable rate submersible centrifugal or positive displacement type pump (i.e., bladder or piston pump) will be used for purging and sampling; however, if the water table is less than 20 ft below ground surface (bgs) a peristaltic pump may be employed as long as the constituents measured are not influenced by negative pressures. The pump and associated tubing used shall be constructed of inert materials and compatible with the parameter(s) to be collected. The placement of the pump intake should be positioned with a calibrated sampling pump hose, sounded with a weighted-tape or using a pre-measured hose. Refer to the available well information to determine the depth and length of the screened interval. The pump should be adequately supported once it has been lowered to ensure that it will not shift during purging. Record the depth of the pump intake after lowering the pump into location. For wells with dedicated pumping equipment, pump depth should be confirmed and equipment condition recorded.
- C. Measure the water level (nearest 0.01 feet) and record the information on the Groundwater Sampling Record and/or in the field notebook. The water level indicator should remain in the well to allow for periodic measurement of the water level during purging.
- D. Connect the discharge line from the pump to a flow-through cell to measure field water quality parameters. If turbidity measurements are to be collected using a separate instrument from that employed to monitor water quality in the flow through cell, a "T" connection is needed prior to the flow-through cell to allow for the collection of water for turbidity measurements. The discharge line from the flow-through cell must be directed to a container to hold the

purge water during the purging and sampling of the well.

- E. Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs. Adjust pump speed until little or no drawdown is evident (less than 0.33 ft). If the minimal drawdown that can be achieved exceeds 0.33 feet but remains stable, continue purging until field parameters stabilize. Typically flow rates should be within 0.1 L/min to 0.5 L/min; however highly productive aquifers may allow for higher flow rates to be used. Adjustments to the flow rate to achieve stabilization should be made as quickly as possible to minimize agitation of the water column. It should be noted that this goal may be difficult to achieve under some circumstances due to geologic heterogeneities within the screened interval, and may require adjustment based on well-specific conditions and site experience.
- F. Measure the discharge rate of the pump using a calibrated discharge volume measurement and stopwatch. Also, measure the water level and record both flow rate and water level on the Groundwater Sampling Record and/or in the field notebook. Continue purging, monitor and record water level and pump rate every three to five minutes during purging.
- G. During purging, a minimum of one tubing volume (including the volume of water in the pump and flow-cell) must be purged prior to recording the water-quality indicator parameters. Then monitor and record the water-quality indicator parameters every three to five minutes. The water-quality indicator field parameters are turbidity, dissolved oxygen, specific conductance, pH, ORP, and temperature. The parameters are considered to have stabilized if on three successive readings of the water quality field parameters meet the following criteria:
- ☐ pH +/- 0.1 S.U.
  - ☐ Specific Conductance 3% difference
  - ☐ Temperature +/- 1°C
  - ☐ ORP +/- 10 mV
  - ☐ Turbidity 10% difference for values greater than 10 NTU
  - ☐ Dissolved Oxygen 10% difference
- H. If a stabilized drawdown in the well can't be maintained at 0.33 feet and the water level is approaching the top of the screened interval, reduce the flow rate or turn the pump off (for 15 minutes) and allow for recovery. It should be noted whether or not the pump has a check valve. A check valve is required if the pump is shut off. Begin pumping at a lower flow rate, if the water draws down to the top of the screened interval again, turn pump off and allow for recovery. If two tubing volumes (including the volumes of water in the field pump and flow-cell) have been removed during purging, then sampling can proceed next time the pump is turned on. This information should be noted in the field notebook or Groundwater Sampling Record.
- I. If specified in the monitoring plan, a clean plastic disposable apron may be placed adjacent to or around the well to prevent equipment and sample containers from coming into contact with surface materials, prior to collecting samples from a well. Alternatively, a clean field table may be set up near the

well. If used, the table will be cleaned (Section 5.1) before and after use at each well.

- J. During sampling, maintain the same pumping rate or reduce slightly for sampling in order to minimize any additional disturbance of the water column. Samples should be collected directly from the discharge port of the pump tubing prior to passing through the flow-cell. The sequence of the sampling is immaterial unless filtered (dissolved) samples are collected which must be done last. All sample containers should be filled with minimal turbulence by allowing the groundwater to flow from the tubing gently down the inside of the container. In the event that the groundwater is turbid (greater than 10 NTUs) a filtered metals sample may be collected. If a filtered metals sample is to be collected, then an in-line filter is fitted at the end of the discharge tubing and the sample is collected after the filter.

Sample bottles that do not contain preservative should be rinsed with the sample water prior to filling.

- K. Remove the pump from the well. Decontaminate the pump and tubing if non-dedicated equipment is used.
- L. Where more than one well within a specific field or site is to be sampled, the sampling sequence should begin with the well having the lowest suspected level of contamination, if known. Successive samples should be obtained from wells with increasing suspected contamination. If the relative degree of suspected contamination at each well cannot be reasonably assumed, sampling should proceed from the perimeter of the site towards the center of the site. The sampling sequence should be arranged such that wells are sampled in order of increasing proximity to the suspected source of contamination, starting from the wells upgradient of the suspected source.
- M. Sampling activity for each monitoring well will be recorded on a Groundwater Sampling Record and the stabilized field parameters may also be recorded in the field notebook.

## 4.2 Volume-Based Sample Collection

In instances where low-flow sampling is not possible based on site-specific conditions (a minimum and stable drawdown cannot be maintained) samples from wells will be collected using a volume-based approach, if the yield of the well is sufficient, as follows:

- A. The depth to water in a well and the total depth will be measured using the procedures discussed in Section 3, above.
- B. A positive displacement pump, submersible pump, and/or bailer will be used for removing the groundwater from the monitoring wells (purgings). Equipment used for purging and sampling may be permanently installed (dedicated) in

the monitoring wells. Care must be taken that bailers and/or tubing are constructed from materials that will not affect the sample analyses. The well pump intake is to be set at the midpoint of the screened interval, unless otherwise specified in the monitoring plan in a manner consistent with that specified for the low-flow sampling above. Pumping is to be performed in such manner as to remove stagnant water while trying to minimize exposing the screened interval to atmospheric conditions and obtain the most representative sample.

- C. Wells will be pumped or bailed until at least the volume of water removed is equal to three well casing volumes (volume of standing water in the well based upon total depth of well, the depth to water, and the well casing diameter). The purge rate must not reach a point where the recharge water is entering the well in an agitated manner (cascading water over the screen interval) and the water level in the well during purging should not be allowed to drop below the pump intake. During pumping, water level measurements will be collected (as described for low-flow sampling) and the purging rate adjusted to ensure that these conditions do not occur.
- D. To ensure that the water samples are representative of the water-yielding zone, periodic measurements of the temperature, pH, dissolved oxygen, ORP, specific conductance and turbidity will be made. A flow-through cell may be used if purging with a pump. Measurements will be recorded for the initial water removed at a minimum following each well volume purged. Note that indicator parameters dissolved oxygen and ORP cannot be accurately measured using discrete samples obtained during bailing (due to exposure to the atmosphere and entrained air becoming trapped in the sampling probe). These parameters will only be collected using a flow-through cell. The sample will be collected only when the indicator parameters have stabilized (as discussed above in Section 4.1). No more than six well volumes should be removed to prevent the effects of over pumping. If the indicator parameters have not stabilized following six well volumes the field instruments will be recalibrated and checked for possible malfunction. If no problems are found, sampling can be conducted; however, the Project Manager will be notified and all information will be recorded in the field notebook and/or Groundwater Sampling Record. If the yield of the well is low such that it can be bailed or pumped dry, then the recharged groundwater in the well will be considered representative regardless of the number of casing volumes of groundwater removed, since all standing water in the well has been replaced by recharge from the water-yielding zone. If a well is purged dry, the well can be sampled upon 90% recovery or after two hours, whichever occurs first.
- E. If specified in the monitoring plan, a clean plastic disposable apron may be placed adjacent to or around the well to prevent equipment and sample containers from coming into contact with surface materials, prior to collecting samples from a well. Alternatively, a clean field table may be set up near the well. If used, the table will be cleaned (Section 5.1) before and after use at each well.
- F. Sample containers prepared specifically for the required analyses by the analytical laboratory or their supplier will be used for sample collection. Glass



sample bottles should be filled to near the top. To account for slight expansion due to temperature changes, leave headspace approximately equivalent to the volume of liquid which would fill the bottle's cap. Plastic sample bottles should be filled completely. Splashing of the water in the sample container and exposure to the atmosphere shall be minimized during sampling. The container cap will be screwed on tightly immediately after filling the sample container. Under this protocol, samples should be collected in order of decreasing volatility (i.e., most volatile samples will be collected first). Sample filtration, if necessary, is discussed in Section 4.5 of this SOP.

Sample bottles that do not contain preservative should be rinsed with the sample water prior to filling.

- G. Where more than one well within a specific field or site is to be sampled, the sampling sequence should begin with the well having the lowest suspected level of contamination, if known. Successive samples should be obtained from wells with increasing suspected contamination. If the relative degree of suspected contamination at each well cannot be reasonably assumed, sampling should proceed from the perimeter of the site towards the center of the site. The sampling sequence should be arranged such that wells are sampled in order of increasing proximity to the suspected source of contamination, starting from the wells upgradient of the suspected source.
- H. Sampling activity for each monitoring well will be recorded on a Groundwater Sampling Record and the stabilized field parameters may also be recorded in the field notebook.

#### **4.3 Non-Purge Sample Collection Using a HydraSleeve™**

In monitoring wells with very low yield and/or where low-flow sampling or volume-based purging is not practical, a non-purge method, sampling using a HydraSleeve™, may be used to collect groundwater samples by the following procedure:

- A. The depth to water in a well and the total well depth will be measured using the procedures discussed in Section 3, above. This measurement can be used to determine the preferred position of the HydraSleeve within the well screen.
- B. Determine the necessary length of HydraSleeves needed for the specific well screen length and/or water column height to recover the necessary sample volume. HydraSleeves are manufactured in standard lengths of 30, 36, and 60 inches. However, up to three 30-inch HydraSleeves may be installed in series on a single tether (using plastic cable ties) to achieve more volume. Alternatively, a TurboSleeve may be used, which is a larger HydraSleeve that allows recovery of more sample volume. Per manufacturer's recommendations, the TurboSleeve should be allowed to equilibrate in the

well for 24 hours before retrieval to allow for full compression of the sleeve for full sample recovery (see step G below).

- C. Measure the correct amount of tether cord needed to suspend the HydraSleeve in the well so the weight will not rest on the bottom of the well and the desired depth is achieved.
- D. Remove the HydraSleeve from its packaging, unfold and hold it by its top. Crimp the top of the HydraSleeve by folding the hard polyethylene reinforcing strips at the holes.
- E. Attach the spring clip to the holes to ensure the top of the HydraSleeve will remain open until the sampler is retrieved. Attach the tether to the spring clip with a strong knot (or tether can be attached to one of the holes at the top of the HydraSleeve).
- F. Fold the flaps with the two holes at the bottom of the Hydra Sleeve together and slide the weight clip through the holes. Attach the weight to the bottom of the weight clip to ensure the HydraSleeve will descend to the desired depth.
- G. To deploy the HydraSleeve, carefully lower the HydraSleeve on its tether to the desired depth in the water column. Hydrostatic pressure will keep the self-sealing check valve at the top of the HydraSleeve closed and ensure that it remains flat and empty and will only fill with groundwater from the desired interval when it is retrieved.
- H. To retrieve the HydraSleeve to collect groundwater samples, pull up the tether 30 to 45 inches (36 to 54 inches for longer HydraSleeves) in one smooth motion at a rate of about one inch per second or faster. This motion will open the top check valve and allow the HydraSleeve to fill. When the HydraSleeve is full, the top check valve will close and the full weight of the HydraSleeve can be felt by the sampler. Continue to pull the HydraSleeve upward to the top of the well to retrieve. Two persons are needed to retrieve a TurboSleeve, if used, due to its length and flexibility.
- I. Once recovered, decant and discard the small volume of water trapped in the HydraSleeve above the top check valve.
- J. To fill sample bottles, remove the discharge tube from its sleeve. While holding the HydraSleeve at the check valve, puncture the HydraSleeve just below the check valve with the pointed end of the discharge tube. Discharge the water into the sample bottles as needed.
- K. Any leftover water from the HydraSleeve can be poured into a separate vessel for the measurement of groundwater field parameters as needed.
- L. Dispose each used HydraSleeve after use at an individual well.

#### **4.4 Non-Purge Sample Collection by Bailer**

In monitoring wells with very low yield where low-flow sampling or volume-based purging is not practical and sampling with HydraSleeves™ is not feasible, sampling by bailer without purging the well may be used to collect groundwater samples.

Sampling by bailer may be used by the following procedure:

- A. The depth to water in a well and the total depth will be measured using the procedures discussed in Section 3, above. This measurement can be used to determine the height of water and the volume of groundwater within the well screen.
- B. A clean, sufficiently weighted PVC or polyethylene bailer will be used attached to a pre-measured length of either coated stainless steel cable or nylon rope tether for each well to be sampled by bailing.
- C. The bailer will be slowly lowered through the water column to the well screen interval on the pre-measured tether. Slow and consistent movement of the bailer downward through the well allows the water within the well to pass through the bailer.
- D. When the desired depth within the well screen interval is reached, the downward movement of the bailer will immediately be reversed and the bailer slowly retrieved to the surface. This action allows the bailer to collect water representative of conditions within the well screen interval while minimizing generation of turbid conditions within the well.
- E. Steel cable or rope will not be allowed to touch the ground surface during retrieval. A reel, tub, tarp, or plastic sheeting can be used to prevent contact with the ground.
- F. Upon retrieval of the bailer, sample bottles for total and dissolved metals analysis will be filled first, followed by the remaining sample bottles for other parameters. If more sample volume is needed, the bailer will again be slowly lowered to the screen interval and retrieved as necessary until required sample bottles have been filled.
- G. If a filtered metals sample is to be collected, the necessary volume can be filtered from one clean, non-preserved sample bottle as needed.
- H. Field parameters will be measured in the instrument cup or another rinsed container following collection of sample bottles. A small aliquot of sample volume will be poured from the bailer for the collection of field parameters.
- I. If the well bails dry but additional sample volume is required, the volume will be removed from the well via bailer if such recharge occurs in the well within 24 hours.

#### **4.5 Sample Filtration**

When required, a field-filtered water sample will be collected using a disposable, in-line 0.45 micron (□m) filter. The water sample will be pumped through the filter attached directly to the discharge tubing. A peristaltic pump and a clean section of Tygon (polyvinylchloride) tubing, silicone tubing, or other appropriate method may be used if the sample is collected via bailer. The filter cartridge will be rinsed according to the manufacturer's recommendations. If there are no recommendations available, for rinsing pass through a minimum aliquot of 100 ml of sample water prior to collection of sample into the containers. Both the filter and tubing will be disposed between samples.

#### **4.6 Sample Containers and Volumes**

The sample containers will be appropriate to the analytical method and will be obtained from the water analysis laboratory or other approved source. Different containers will be required for specific groups of analytes in accordance with USEPA Methods, project-specific requirements, and/or other local jurisdictional guidance. The sampler will confirm with the laboratory performing the analyses that appropriate bottleware and preservatives are used and ensure that a sufficient volume of sample is collected.

#### **4.7 Sample Labeling**

Sample containers will be labeled with self-adhesive tags. Each sample will be labeled with the following information using waterproof ink:

- ☐ Project identification;
- ☐ Lab Name;
- ☐ Sample identification;
- ☐ Date and time samples were obtained;
- ☐ Matrix;
- ☐ Requested analyses and method;
- ☐ Bottle type;
- ☐ Treatment (preservative added, filtered, etc.);
- ☐ Lab QC (if applicable); and
- ☐ Initials of sample collector(s).

#### **4.8 Sample Preservation and Storage**

If required by the project or analytical method, water samples submitted for chemical analysis will be stored at 4 °C in ice-cooled, insulated containers immediately after collection. Preservation and storage methods depend on the chemical constituents to be analyzed and should be discussed with the laboratory prior to sample collection. USEPA and/or other local jurisdictional requirements and/or the requirements of a project-specific plan (e.g., sampling and analysis plan, work plan, quality assurance project plan, etc.) shall be adhered to in preservation and storage of water samples.

#### **4.9 Sample Custody**

Samples shall be handled and transported according to the sample custody procedures discussed in SOP No. 2 (SAMPLE CUSTODY, PACKAGING, AND SHIPMENT). Sampling personnel shall document each sample on the Chain-of-Custody Record

#### **4.10 Field Measurements**

Specific conductance, pH, dissolved oxygen, ORP, temperature, and turbidity measurements will be performed on water samples at the time of sample collection. The only exceptions will be for DO and ORP when the samples are collected via bailer or in those instances where a flow-through cell cannot be used. Data obtained from these (or other) field water quality measurements will be recorded on the appropriate sampling records or in the field notebook. Separate aliquots of water shall be used to make field measurements (i.e., sample containers for laboratory analysis shall not be reopened).

For groundwater samples, field measurement intervals will be as presented above. If the parameters have not stabilized, check to insure that the field instruments are operating correctly and remain calibrated. Recalibrate the instruments if needed, if an instrument cannot be calibrated it will be labeled needing repair and removed from service. Field measurements and purging will continue until three consecutive readings have stabilized to within the following limits or until a maximum of six casing volumes have been removed:

- pH +/- 0.1 S.U.;

- ☐ Specific Conductance 3% difference;
- ☐ Temperature +/- 1°C;
- ☐ ORP +/- 10 mV;
- ☐ Turbidity 10% difference for values greater than 10 NTU; and
- ☐ Dissolved Oxygen 10% difference.

#### **4.10.1 Temperature Measurement**

Temperature will be measured directly from the water source or from a separate sample aliquot. Temperature measurements will be made with a mercury-filled thermometer, bimetallic-element thermometer, or electronic thermistor. All measurements will be recorded in degrees Celsius (°C). When a flow-through cell is used the temperature can be measured directly via a multi-parameter instrument as per the manufacturer's instructions.

#### **4.10.2 pH Measurement**

A pH measurement will be made by dipping the probe directly into the water source or into a separate sample aliquot. Prior to measurement, the container in which the field parameter sample will be collected will be acclimated to the approximate temperature of the sample. This can be accomplished by immersing the container in water removed from a well during the purging process. The pH measurement will be made as soon as possible after collection of the field parameter sample, preferably within a few minutes, using a pH electrode. The value displayed on the calibrated instrument will be recorded after the reading has stabilized. If the value falls outside of the calibrated range, then the pH meter will be recalibrated using the appropriate buffer solutions. When a flow-through cell is used, the pH can be measured directly via a multi-parameter instrument as per the manufacturer's instructions.

#### **4.10.3 Dissolved Oxygen**

Dissolved oxygen (DO) will be measured by using a suitable multi-parameter meter that can be placed into a flow-through cell and sealed such that exposure to the atmosphere is prevented. DO measurements will be reported in milligrams per liter (mg/L). The instrument will be calibrated in accordance with SOP No. 31 (WATER QUALITY METER CALIBRATION).

#### 4.10.4 Oxidation Reduction Potential

Oxidation Reduction Potential (ORP) will be measured by using a suitable multi-parameter meter that can be placed into a flow-through cell and sealed such that exposure to the atmosphere is prevented. ORP measurements will be reported in mV. The instrument will be calibrated in accordance with SOP No. 31 (WATER QUALITY METER CALIBRATION).

#### 4.10.5 Specific Conductance Measurement

Specific conductance will be measured by dipping the probe directly into the water source or into a separate sample aliquot. The probe must be immersed to the manufacturer's recommended depth. Specific conductance will be reported in micromhos/cm or microsiemens/cm at 25 °C. If the meter is not equipped with an automatic temperature compensation function, then the field value will be adjusted at a later time using the temperature data and the following formula:

$$SC_{25} = SC_T / [1 + \{(T - 25) \times 0.025\}]$$

where:  $SC_{25}$  = specific conductance at 25 °C  
 $SC_T$  = specific conductance measured at temperature T (°C)  
T = sample temperature (°C)

The value displayed on the calibrated instrument will be recorded after the reading has stabilized. If the value falls outside of the calibrated "range" set by the range dial on the instrument, then the range setting will be changed to a position that gives maximum definition. If the specific conductance value falls outside of the calibrated range of the conductivity standard solution, then the instrument will be recalibrated using the appropriate standard prior to measurement. When a flow-through cell is used the specific conductance can be measured directly via a multi-parameter instrument as per the manufacturer's instructions.

#### **4.10.6 Turbidity**

Turbidity will be measured by using a field portable nephelometer separate from the multi-parameter meter used for DO and ORP and capable of reading down to 0.1 NTU. Turbidity will be measured directly from the water source or from a separate sample aliquot. The instrument will be calibrated at least daily prior to initiating field activities and periodically throughout the day or as recommended by the instrument manufacturer. Turbidity measurements will be reported in nephelometric turbidity units (NTU). When a flow-through cell is used, the turbidity can be measured directly via a multi-parameter (e.g., YSI Sonde 6920) instrument, if so equipped, as per the manufacturer's instructions.

#### **4.10.7 Equipment Calibration**

Equipment used to measure field parameters will be calibrated daily in the field in accordance with SOP No. 31 (WATER QUALITY METER CALIBRATION) prior to any measurements being taken.

### **5.0 DOCUMENTATION**

#### **5.1 Groundwater Sampling Record**

Each sampling event for each monitoring well will be recorded on a Groundwater Sampling Record form (which may be in paper or electronic format) or in the field notebook.

The documentation should include the following:

- ☐ Project identification;
- ☐ Location identification;
- ☐ Sample identification(s) (including quality control samples);
- ☐ Date and time of sampling;
- ☐ Purging and sampling methods;
- ☐ Sampling depth;
- ☐ Name(s) of sample collector(s);
- ☐ Inventory of sample bottles collected including sample preservation (if any), number, and types of sample bottles;
- ☐ Total volume of water purged;



- ☐ Results of field measurements and observations (time, cumulative purge volume, temperature, pH, specific conductance, turbidity, sediment, color, purge rate);
- ☐ Equipment cleaning record;
- ☐ Description and identification of field instruments and equipment; and
- ☐ Equipment calibration record; and
- ☐ Number of photos (if any were taken).

When the sampling activity is completed, the Groundwater Sampling Record (whether in print or electronic format) will be checked by the Project Manager or his/her designee, and the original record will be placed in the project file.

## **6.0 QUALITY ASSURANCE/QUALITY CONTROL**

### **6.1 Equipment Decontamination/Cleaning**

Steel surveyor's tapes, electric well probes, and other measuring tapes shall be cleaned prior to use and after measurements in each well are completed. Cleaning shall be accomplished by either: (1) washing with a laboratory-grade detergent/water solution, rinsing with clean, potable, municipal water, final rinsing with distilled or deionized water, or (2) steam cleaning followed by rinsing with distilled or deionized water.

Sample bottles and bottle caps will be pre-cleaned and prepared by the analytical laboratory or their supplier using standard USEPA-approved protocols. Sample bottles and bottle caps will be protected from dust or other contamination between time of receipt by the sampler(s) and time of actual usage at the sampling site.

Groundwater sampling equipment may be dedicated to a particular well at a project site. Prior to installation of this equipment, all equipment surfaces that will be placed in the well or may come in contact with groundwater will be cleaned to prevent the introduction of contaminants.

Sampling equipment that will be used at multiple wells or sampling locations will be cleaned after sampling at each location is completed in accordance with the SOP entitled EQUIPMENT DECONTAMINATION (SOP No. 7).

Equipment such as submersible electric pumps, which cannot be disassembled for cleaning, will be cleaned by circulating a laboratory-grade detergent (e.g., Alconox) and potable water solution through the assembly, followed by clean potable water from a municipal supply, and then by distilled or deionized water. Equipment cleaning methods will be recorded on the Groundwater Sampling Record.

## 6.2 Technical and Records Reviews

The Project Manager or designated reviewer will check and verify that documentation has been completed and filed per this procedure.

In addition, all calculations of water-level elevations must be reviewed before they are submitted to the project file and used to describe site conditions. The calculation review should be performed by technical personnel familiar with this procedure. Evidence of the completed review and any necessary corrections to calculations should also be submitted to the project file.

## 7.0 REFERENCES

- U.S. Environmental Protection Agency (USEPA), Region 9. Standard Operating Procedure for the Standard/Well-Volume Method for Collecting a Ground-Water Sample from Monitoring Wells for Site Characterization. Available online at: [www.epa.gov/region9/qa/pdfs/finalgwsamp\\_sop.pdf](http://www.epa.gov/region9/qa/pdfs/finalgwsamp_sop.pdf).
- USEPA, 2010. Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells. \_EQASOP-GW-001 Region 1 Low-Stress (Low-Flow) SOP, Revision Number 3, July 30, 1996, Revised January 19, 2010. <http://www.epa.gov/region1/lab/qa/pdfs/EQASOP-GW001.pdf>
- USEPA, 2002, Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers, Ground Water Forum Issue Paper. U.S Environmental Protection Agency, Technology Innovative Office, Office of Solid Waste and Emergency Response, Washington D.C., Douglas Yeskis, EPA Region 5, Chicago IL, and Bernard Zavala, EPA Region 10, Seattle, WA, EPA 542-S-02-001. May 2002. [http://www.epa.gov/superfund/remedytech/tsp/download/gw\\_sampling\\_guide.pdf](http://www.epa.gov/superfund/remedytech/tsp/download/gw_sampling_guide.pdf)
- U.S. Geological Survey (USGS), variously dated. National Field Manual for the Collection of Water-Quality Data: U.S. Geological Survey Techniques of Water-Resources Investigations, Book 9, Chapters A1-A9. Available online at <http://pubs.water.usgs.gov/twri9A>.

## **SOP No. 5 Surface Water Sampling**

## **STANDARD OPERATING PROCEDURE No. 5**

### **SURFACE WATER SAMPLING**

#### **1.0 SCOPE AND APPLICABILITY**

This Standard Operating Procedure (SOP) describes the protocol to be followed during sampling of surface water. The procedures presented herein are intended to be general in nature and are applicable when referenced by site-specific or project-specific planning documents. Appropriate modifications to the procedures may be made to accommodate project-specific protocols when approved in writing or via email by the Project Manager.

The objectives of the water quality sampling procedures are to minimize changes in surface water chemistry during sample collection and to maximize the probability of obtaining a representative, reproducible surface water sample.

#### **2.0 BASIS FOR METHODOLOGY**

The methods and procedures described in this SOP were developed from these sources:

- U.S. Geological Survey (USGS) (variously dated) National Field Manual for the Collection of Water-Quality Data: U.S. Geological Survey Techniques of Water-Resources Investigations, Book 9, Handbooks for Water-Resources Investigations, Chapters A1-A9.
- U.S. Environmental Protection Agency (USEPA) (2003) USEPA Region 8 SOP EH-#1 Technical Standards Operating Procedure – Surface Water Sampling East Helena Site, Montana.

#### **3.0 PROCEDURES**

##### **3.1 Sample Collection**

Individual samples from surface water sampling stations will be collected as follows:

- A. Where multiple sampling stations exist along a moving water source (i.e., a creek or drainage channel), the downstream station will be sampled first. A moving water sample will be taken from the portion of the water with maximum flow at any given sampling station unless otherwise specified. If the sampling point is inaccessible from shore, the sampling personnel will enter the water from a point downstream of the sampling point, taking care not to disturb the water.
- B. A standing water sample will be taken at a point in the body of water at least three feet from the shore, if possible, or unless otherwise specified.
- C. A surface water sample will be collected according to one of the following, or similar, techniques.
  - 1. Direct Method -- Sample bottle or disposable container (e.g., cubitainer) is uncapped and inverted, submerged to the specified depth, turned upright pointing upstream, removed from the water, and then capped (if actual sample bottle used). Add preservative, if any, after sample collection.
  - 2. Dipper Method -- Sample bottle or container attached to a pole is dipped in the water, raised above the water, and then capped (if actual sample bottle used).
  - 3. Bailer Method -- An appropriate sampling bailer with a ball check valve is submerged to the desired sample depth, either directly or by suspending the bailer on a rope from a pole.
  - 4. Syringe Method (for very shallow water) -- A disposable plastic filtering syringe may be used to collect very shallow surface water without disturbing the sediment. The syringe will be disposed of after each use.
  - 5. Peristaltic Pump Method -- The sample is collected through a section of new, clean, flexible Tygon (polyvinylchloride) or silicone tubing. The tubing intake will be secured manually or by attaching weights. This procedure may be modified to collect the sample through a Teflon tube into a sample flask by running the pump on a vacuum.
  - 6. Kemmerer Bottle -- Use a properly decontaminated Kemmerer bottle. Set the sampling device so that the upper and lower stoppers are pulled away from the body, allowing water to enter the tube. Lower the pre-set sampling device to the predetermined depth. Avoid disturbing the bottom. Once at the required depth, send the weighted messenger down the suspension line, closing the device. Retrieve the sampler and discharge the first 10-20 mL from the drain to clear water that may not be representative of the sample. Repeat as needed to collect the needed volume.

7. Van Dorn Sampler -- Set the device so that the end stoppers are pulled away from the body allowing surface water to enter the tube. Lower the sampler to the predetermined depth. Once at the required depth send the weighted messenger down the suspension line, closing the sampling device. Retrieve the sampler and decant the first 10- 20 mL from the drain to clear water that may not be representative of the sample from the valve. Repeat as needed until the required volume to fill sample bottles is collected.
  8. Bacon Bomb Sampler -- Lower the bacon bomb sampler carefully to the desired depth, allowing for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line until taut. This will allow the sampler to fill. Release the trigger line and retrieve the sampler. Decant the first 10-20 mL from the drain to clear water that may not be representative of the sample from the valve. Repeat as needed until the required volume to fill sample bottles is collected.
- D. For the direct and dipper methods, the mouth of the sample collection container will be oriented downstream. The first collected water will be used to rinse the sampling equipment. Sample bottles that do not contain preservative should be rinsed with the sample water prior to filling. Subsequent water collected will be used to fill the analytical sample bottles until all bottles are filled. Field measurement of parameters will be taken once for each sampling station at the time of sample collection. Field parameters (pH, specific conductance, temperature, dissolved oxygen (DO), oxidation-reduction potential (ORP), odor, turbidity, and/or sediment) will be measured from a separate container (instruments will not contact the analytical samples) or directly from the water body sampled downgradient of the sample collection location.
- E. To mark the exact sampling location, either: (1) a stake or pole identifying the sampling station should be placed at or near the sampling station for future identification of the location, or (2) the sample location will be recorded in a Global Positioning System (GPS) device and coordinates will be downloaded at the end of the field event. Personnel will record a brief description of the stake or pole location in relation to permanent landmarks, and the sampling location in relation to the stake or pole (example: stake is approximately 100 feet west along Markley Creek from Somersville Road, on north-side shore. Sampling point is 25 feet south of stake, in middle of Markley Creek), or the GPS coordinates of the location. Personnel will include a sketch map of the sampling station in the Surface Water Sampling Record (attached form or similar).

### 3.2 Sample Filtration

When required, a field-filtered water sample will be collected using a disposable, in-line 0.45 micron ( $\mu\text{m}$ ) filter. The water sample will be pumped through the filter attached

directly to the discharge tubing. A peristaltic pump and a clean section of Tygon (polyvinylchloride) tubing, silicone tubing, or other appropriate method may be used. The filter cartridge will be rinsed according to the manufacturer's recommendations. If there are no recommendations for rinsing, pass through a minimum aliquot of 100 ml of sample water prior to collection of sample into the containers. Both the filter and tubing will be disposed between samples.

### **3.3 Sample Containers and Volumes**

The sample containers will be appropriate to the analytical method and will be obtained from the water analysis laboratory or other approved source. Different containers will be required for specific groups of analytes in accordance with U.S. EPA Methods, project-specific requirements, and/or other local jurisdictional guidance. The sampler will confirm with the laboratory performing the analyses that appropriate bottleware and preservatives are used and ensure that a sufficient volume of sample is collected.

### **3.4 Sample Preservation and Storage**

If required by the project or analytical method, water samples submitted for chemical analysis will be stored at 4°C in ice-cooled, insulated containers immediately after collection. Preservation and storage methods depend on the chemical constituents to be analyzed and should be discussed with the water analysis laboratory prior to sample collection. EPA and/or other local jurisdictional requirements and/or the requirements of a project-specific plan (e.g., sampling and analysis plan, work plan, quality assurance project plan, etc.) shall be adhered to in preservation and storage of water samples.

### **3.5 Field Measurements**

Specific conductance, pH, turbidity, DO, ORP, and temperature measurements will be performed on water samples at the time of sample collection. Data obtained from these (or other) field water quality measurements will be recorded on the appropriate sampling records and/or in the field notebook. Separate aliquots of water shall be used to make field measurements (i.e., sample containers for laboratory analysis shall not be

reopened). For surface water sampling, the parameters will be measured once and recorded.

### **3.5.1 Temperature Measurement**

Temperature will be measured directly from the water source or from a separate sample aliquot. Temperature measurements will be made with a mercury-filled thermometer, bimetallic-element thermometer, multiprobe, or electronic thermometer. All measurements will be recorded in degrees Celsius (°C) after the reading has stabilized.

### **3.5.2 Turbidity**

Turbidity will be measured by using a field portable nephelometer or integrated turbidity sensor on a multi-parameter meter (e.g., YSI Sonde 6920) capable of reading down to 0.1 NTU. The instrument will be calibrated at least daily or according to the manufacturer's recommendations prior to initiating field activities and periodically throughout the day or as recommended by the instrument manufacturer. Turbidity measurements will be reported in nephelometric turbidity units (NTU).

### **3.5.3 pH Measurement**

A pH measurement will be made by dipping the probe directly into the water source or into a separate sample aliquot. Prior to measurement, the container in which the field parameter sample will be collected will be acclimated to the approximate temperature of the sample. This can be accomplished by immersing the container in water collected from the sampling location. The value displayed on the calibrated instrument will be recorded after the reading has stabilized. If the value falls outside of the calibrated range, then the pH meter will be recalibrated using the appropriate buffer solutions.

### **3.5.4 Specific Conductance Measurement**

Specific conductance will be measured by dipping the probe directly into the water source or into a separate sample aliquot. The probe must be immersed to the manufacturer's recommended depth. Specific conductance will be reported in



micromhos/cm at 25 °C . If the meter is not equipped with an automatic temperature compensation function, then the field value will be adjusted at a later time using the temperature data and the following formula:

$$SC_{25} = SC_T / [1 + \{(T - 25) \times 0.025\}]$$

where:  $SC_{25}$  = specific conductance at 25 °C  
 $SC_T$  = specific conductance measured at temperature T (°C)  
T = sample temperature (°C)

The value displayed on the calibrated instrument will be recorded after the reading has stabilized. If the value falls outside of the calibrated "range" set by the range dial on the instrument, then the range setting will be changed to a position that gives maximum definition. If the specific conductance value falls outside of the calibrated range of the conductivity standard solution, then the instrument will be recalibrated using the appropriate standard prior to measurement.

### 3.5.5 Dissolved Oxygen

Dissolved oxygen measurements taken from surface water locations should be representative of the conditions being monitored. For example, if a sample is to be collected from the middle of a stream cross-section, that is also where the DO measurement should be taken.

The dissolved oxygen probe should be fully immersed in the water body being monitored. If the DO measurement is taken from a stream, the probe should be elevated above the stream bed to minimize disturbance of channel sediments. If DO measurements are taken from a water body that is not flowing, the probe should be slowly raised and lowered so that water is moving past the DO probe membrane.

Dissolved oxygen readings should be recorded after allowing sufficient time for the probe to equilibrate and the readings to stabilize. For surface water measurements, the DO reading will typically stabilize as soon as the probe has equilibrated. The value displayed on the calibrated instrument will be recorded after the reading has stabilized.

### **3.5.6 Oxidation-Reduction Potential**

Oxidation-Reduction potential will be measured by dipping the probe directly into the water source or into a separate sample aliquot. The probe must be immersed to the manufacturer's recommended depth. ORP will be reported in mV. Readings should be recorded after allowing sufficient time for the probe to equilibrate and the readings stabilize.

### **3.5.7 Equipment Calibration**

Equipment used to measure field parameters will be calibrated in the field daily in accordance with SOP No. 31 (WATER QUALITY METER CALIBRATION) by field personnel prior to the collection of any samples

## **4.0 DOCUMENTATION**

Each sampling event for each surface water sampling station will be recorded in the field notebook and/or on a separate Surface Water Sampling Record form (in print or electronic format – see attached). The documentation should include the following:

- A. Project identification;
- B. Location identification (sampling station);
- C. Sample identification(s) (including quality control samples);
- D. Date and time of sampling;
- E. Weather conditions;
- F. Description of sampling location;
- G. Sampling method;
- H. Description of flow measurement method, if applicable, and any flow data;
- I. Instrument calibration and cleaning record;
- J. Results of field measurements and observations (time, temperature, pH, specific conductance, turbidity, DO, ORP);
- K. Name(s) of sample collector(s);

- L. Sketch map showing location of sampling station and permanent landmarks and/or GPS coordinates (if GPS coordinates do not already exist for the sampling station); and
- M. Number of photos (if taken).

When the sampling activity is completed, the Surface Water Sampling Record will be checked by the Project Manager or his/her designee, and the original record will be placed in the project file.

## **5.0 QUALITY CONTROL**

### **5.1 Equipment Decontamination/Cleaning**

Sample bottles and bottle caps will be cleaned and prepared by the analytical laboratory or their supplier using standard EPA-approved protocols. Sample bottles and bottle caps will be protected from dust or other contamination between time of receipt by the sampling personnel and time of actual usage at the sampling site.

Sampling equipment that will be used at multiple sampling locations will be cleaned after sampling at each location is completed in accordance with the SOP No. 7 (EQUIPMENT DECONTAMINATION).

Equipment such as submersible electric pumps, which cannot be disassembled for cleaning, will be cleaned by circulating a laboratory-grade detergent (e.g., Alconox) and potable water solution through the assembly, followed by clean potable water from a municipal supply, and then by distilled or deionized water. Equipment cleaning methods will be recorded in the field notebook and/or on the Surface Water Sampling Record.

### **5.2 Records Review**

The Project Manager or designated reviewer will check and verify that documentation has been completed and filed per this procedure.

## 6.0 REFERENCES

- U.S Environmental Protection Agency (USEPA), 2003. USEPA Region 8 Technical Standard Operating Procedure, Surface Water Sampling (SOP #EH-001, East Helen Site, Montana). September 2003. Adapted from ERT/REAC SOP 2013 Rev 1.0. Available online at [http://nctc.fws.gov/resources/course-resources/pesticides/Monitoring/Surface%20Water%20Sampling\\_EPA.pdf](http://nctc.fws.gov/resources/course-resources/pesticides/Monitoring/Surface%20Water%20Sampling_EPA.pdf)
- U.S. Geological Survey (USGS), variously dated. National Field Manual for the Collection of Water-Quality Data: U.S. Geological Survey Techniques of Water-Resources Investigations, Book 9, Handbooks for Water-Resources Investigations, Chapters A1-A9. Available online at <http://pubs.water.usgs.gov/twri9A>.

<b>SURFACE WATER SAMPLING RECORD</b>					LOCATION ID: _____		
Date: _____ Time: _____ Weather: _____ Page _____ of _____ Weather Past 48 hours: _____ Personnel: _____ Location Description: _____ Water Body Type: _____ Water Present (Y/N) _____ Depth _____ Flow Meas (Y/N) _____ Reason if N _____							
<b>QUALITY ASSURANCE</b>							
Sampling Equipment: _____ Decontamination: Alconox, Distilled Water, Rinse Method of Sampling: _____							
<b>FIELD PARAMETER INSTRUMENTS</b>							
<b>pH Meter:</b> Model: YSI-556 Calibration: 4.00/7.00 pH Buffers After Calibration Meter Read: _____ <b>Conductivity Meter:</b> Model: YSI-556 Calibration: 1,413 $\mu$ S/cm Conductivity Standard After Calibration Meter Read: _____ <b>Temperature Meter:</b> Model: YSI-556 <b>ORP Meter:</b> Model: YSI-556 Calibration: YSI Zobell Solution <b>Turbidity Kit:</b> Model: HF Scientific MicroTPW Turbidity Meter; Calibration: _____							
<b>SAMPLING MEASUREMENTS</b>							
Sample Collection Time	Depth (ft)	pH	Specific Conductance ( $\mu$ mos/cm)	Temp. ( $^{\circ}$ C)	Dissolved Oxygen (mg/L)	Turbidity (NTU)	ORP (mV)
<b>SAMPLE INVENTORY</b>							
Sample Type (circle one):      Primary Sample                      Duplicate                      Equipment Rinsate Blank  SAMPLE ID: _____							
Sample Processing		Container Type	Volume (mL)	Number of Bottles	Filtered	Preservative	Comments
Date	Time						
<b>MAP / COMMENTS</b>							
UTM Coordinates (NAD83):          							
SIGNATURE: _____							

## **SOP No. 6 Surface Water Discharge Measurement**

## **STANDARD OPERATING PROCEDURE No. 6**

### **SURFACE WATER DISCHARGE MEASUREMENT**

#### **1.0 SCOPE AND APPLICABILITY**

This Standard Operating Procedure (SOP) describes the protocol for collecting discharge measurements in streams, ditches, springs, seeps, etc. The procedures presented herein are intended to be general in nature and are applicable when referenced by site-specific or project-specific planning documents. Appropriate revisions may be made to accommodate site-specific conditions or project-specific protocols when approved in writing or via email by the Project Manager.

The objective of this SOP is to provide a consistent method for describing a current, and three methods for measuring discharge: (1) volumetric method, (2) area-velocity method, and (3) flume method. Discharge is defined as the volume rate of water flow, including any substances suspended or dissolved in the water. Discharge will be expressed in cubic feet per second (cfs) or gallons per minute (gpm). Note that the protocol for collection of surface water samples is included in SOP No. 5 (WATER QUALITY SAMPLING).

#### **2.0 BASIS FOR METHODOLOGY**

The methods and procedures described in this SOP were developed from these sources:

- ☐ Rantz, S.E. et al. (1982) Measurement and Computation of Streamflow: Volume 1, Measurement of Stage and Discharge, U.S. Geological Survey Water Supply Paper 2175. U.S. Government Printing Office, Washington, D.C.
- ☐ U.S. Department of the Interior Geological Survey (USGS) (1977) National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, U.S. Geological Survey, Reston, VA.
- ☐ U.S. Environmental Protection Agency (USEPA) (1986) Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual. Environmental Services Division, Region IV, Athens, GA.

### 3.0 PROCEDURES

The selection of an appropriate method for discharge measurement depends on the flow conditions. In some conditions, the flow measurement methods described here may be impossible to implement (e.g., extreme high-flow conditions). If flow conditions cannot be measured at a specific location, then field personnel will attempt to measure flow at a point upstream or downstream of the sample site. The field personnel will also note the conditions that inhibited more accurate measurement at the designated measurement location.

#### 3.1 Current Description

In cases where a discharge measurement is not required but a description of the direction and relative rate of flow is useful, the following method for current measurement may be used.

The current within a moving body of water and its direction is variable by location and depth. Current measurement may be used to define the movement of water at a specified location and depth where a sample is collected. Qualitative measurement of current is made by using a strip of soft tape or cloth attached to the end of a pole. The strip will indicate the presence of water flow and direction of flow at the location and depth. The diameter of the pole should be sufficiently small to prevent directional error. Quantitative measurements may be made using a current meter, which determines the water velocity (feet per second) from pressure exerted by the water, or one of the other methods described in Section 3.4.

#### 3.2 Discharge Measurement

The selection of discharge measurement method depends on stream flow rate and/or specific channel characteristics. For pipes, drain system outfalls, and cases where flows are too small or stream gradients are too high, the volumetric method is appropriate. In cases where water depth is greater than 0.3 feet or the channel cross section is wide, discharge should be measured using the area-velocity method. Where flows are below the practical limit that can be measured with the area-velocity method, the flume method is best.



Where the total discharge is conveyed through two channels or differing types, a combination of these methods may be appropriate.

### **3.3 Volumetric Method**

The volumetric method is a simple and accurate method for measuring flow from small discharges such as gravity flow discharged from pipe outlets. This method involves observing the time required to fill or partly fill a calibrated container to a known volume. Alternatively, in the case of measuring discharge remotely in a sump or standpipe setting, the volumetric method may be performed by capturing flow in a container for a set period of time, no less than 10 seconds, if possible. This volume of water is then measured and discharge is determined.

#### **3.3.1 Equipment**

The volumetric method is particularly useful for the measurement of small flows. Equipment required to make this measurement includes a calibrated container and a stopwatch. Calibrated containers of varying sizes include: 5-gallon bucket, 2-liter graduated cylinder, 1-liter graduated cylinder, 1-liter bucket, etc. Extension rods will be used to hold a container for capturing flow in enclosed areas containing discharging pipes.

#### **3.3.2 Maintenance and Calibration Procedures**

Graduated cylinders are incremented in terms of milliliters and can be easily converted to gallons. The incremental volume of a 5-gallon bucket can be determined by adding known volumes of water and recording the depth after each addition.

#### **3.3.3 Field Procedures**

Upon arrival at the site, the field personnel will evaluate the flow conditions to select the appropriate method for flow measurement. If the flow conditions meet those outlined in Section 3.3, then field personnel will observe and use judgment in approximating the flow volume and will select an appropriately sized volumetric container to use the volumetric method of flow measurement.

Field personnel will use a stopwatch to measure the time required to fill a volumetric container. Field personnel will time flow into the container for a minimum of 10 seconds, if possible. A minimum of five consecutive measurements will be made and noted, and the results will be averaged to determine the discharge.

If remote measurement is necessary, a container will be attached to an extension rod. The field personnel will time flow for a minimum of 10 seconds, if possible. The volume of water will then be poured into a calibrated container, measured, and recorded. A minimum of five such measurements will be made, noted, and the results averaged to determine the discharge.

#### 3.3.4 Discharge Calculations

Discharge will be determined initially in gallons per second (g/s) or in milliliters per second (ml/s). These values will be noted, but the averaged value will be reported in cubic feet per second (cfs). Calculations will be performed as follows:

- ☐ Record each of the measurements in terms of gallons per second or milliliters per second, depending on the volumetric container.
- ☐ If one of the measurements is different from the other measurements by 50 percent or more, then this value will not be used. Instead, five additional measurements will be taken and, provided that none of these measurements differs by greater than 50 percent from the other measurements, these values will be used.
- ☐ Calculate the average of the measured values.
- ☐ Leakage around the discharge pipe, if any, will also be estimated and noted.
- ☐ Convert the averaged value to cfs as follows:
  - to convert ml/s to cfs, multiply by  $3.53 \times 10^{-5}$
  - to convert gal/s to cfs, multiply by 0.134
- ☐ Record discharge in cfs.

### 3.4 Velocity-Area Method

A vertical axis current meter (e.g., March-McBirney Model 2000 Flo-Mate, Hach FH950, Son-Tek, or equivalent) may be used to perform velocity-area method discharge measurements. These instruments use electromagnetic induction to determine the velocity of water. As water flows over a sensor it changes the voltage within the sensor. The changing voltage is processed by the instrument which presents the output as a linear measurement of velocity. Vertical axis current meters can be used at any depth greater than 0.15 feet.

#### 3.4.1 Introduction

The current meter measures velocity in flowing water at a point. The velocity-area method requires measurement of the mean velocity in selected subsections of the stream cross-section. By dividing the stream width into subsections, discharge becomes the total of discharges measured in each subsection. Velocity (V) is measured at each subsection, and discharge becomes the sum of the products of each velocity point and the cross-sectional area of each subsection:

$$Q = \sum(A_i * V_i)$$

where: Q = Streamflow in cfs,  
 A = Area of stream subsection in square feet, and  
 V = velocity in feet per second.

A cross section is defined by the depths at vertical points (# 1, 2, 3, ...n) where the average velocity is measured.

In general, the person(s) measuring discharge should strive to measure no more than 5 percent of the flow in any one subsection (for small streams this is often not practical). The person(s) measuring discharge should divide the channel cross-section into subsections. The total number of subsections (segments) should be large enough to ensure no more than 10 percent of the total flow is contained in any one segment (preferably 5 percent). The location of the metering sites need not be equally spaced across the stream. The

locations should be more closely spaced where water depth or velocity are changing most rapidly or where the channel and flow are more irregular.

Velocities will be measured by the current meter at each measuring point, and each velocity reading will be recorded when the meter stabilizes.

### 3.4.2 Required Measurement Conditions

To make an area-velocity discharge measurement, the following conditions are required:

1. The stream must be channelized or contain relatively straight sections upstream of and at the measurement location.
2. Depth must be greater than 0.2 foot across most of the crosssection being measured.

The ideal channel cross-section is trapezoidal in shape, completely smooth in boundary materials, and possesses a uniform velocity distribution. Such an ideal condition is rarely observed. Therefore, minor modifications to the stream channels will be used to optimize measurement conditions. These modifications may include removal of aquatic vegetation, ice, and moving small stones that impact velocity upstream or downstream of the cross-section. However, no modifications should be made while measurements are being taken.

If flow conditions permit, current meter measurements will be made by wading. Do not switch from one meter to another in the middle of a discharge measurement. The person(s) measuring discharge should stand downstream at arm's length to the side of the flow sensor.

Under open channel laminar flow conditions, the effect of fluid contact with the bed of a stream channel and the air is a vertical distribution of velocities. Consistent with this velocity distribution, actual observation and mathematical theory has demonstrated that a single measurement of velocity taken at 0.6-depth or the average of two point velocities taken at 0.2 and 0.8 of the depth below the surface accurately results in mean velocity in the vertical (USGS Water-Supply Paper 2175, pages 133-134).

If the stream is generally less than 2.5 feet deep, the six-tenths (0.6) method will be used. If the stream is generally greater than 2.5 feet, the two- and eight-tenths (0.2 and 0.8) method, also known as the two-point method, will be used. A complete discussion concerning how to set the wading rod to place the current meter at proper depths is contained in Section 3.4.5, Field Procedures.

In the 0.6-depth method, an observation of velocity made in the vertical at 0.6 of the depth below the surface is used as the mean velocity in the vertical. In the two-point method of measuring velocities, observations are made in each vertical at 0.2 and 0.8 of the depth below the surface. The average of the two observations is taken as the mean velocity in the vertical.

A depth of 1.25 feet will accommodate the 0.6-depth method without causing the meter to be set closer than 0.5 feet from the stream bed; if the meter is set any closer to the stream bed, it will under-register the velocity.

### 3.4.3 Equipment

Current meters and depth and width measuring devices are needed for measurement of discharge. The equipment includes:

- ☐ Top-setting wading rod and current meter
- ☐ Width-measuring devices, either engineer's tape or tagline
- ☐ Stakes for width-measuring devices
- ☐ Calculator

*Top-Setting Wading Rod.* The depth-measuring device that will be used is the wading rod. The current meter is attached to the wading rod. The top-setting wading rod has a 2-inch hexagonal main rod for measuring depth and a 3/8-inch diameter round rod for setting the position of the current meter.

*Current Meter.* Vertical axis current meter, for example, Marsh-McBirney Model 2000 Flo-Mate, Hach FH950, or Son-Tek.

*Engineer's Tape or Tagline.* Tape measures or premarked taglines are used for stream width measurements. Orientation normal to the flow patterns of the stream and elimination of most of the sag, through support or tension, are recommended for improved accuracy.

#### 3.4.4 Maintenance and Calibration Procedures

The Marsh-McBirney Model 2000 Flo-Mate and Hach FH950 will have a zero check performed on the sensor to ensure accurate readings are obtained. First clean the sensor. Then place the sensor in a five gallon bucket of water. Keep it at least three inches away from the sides and bottom of the bucket. To make sure the water is not moving, wait 10 to 15 minutes after positioning the sensor and before taking any zero readings. If needed, follow the manufacturer recommendations to zero adjust the sensor.

To ensure reliable observations of velocity, it is necessary that the current meter be kept in good condition. Before and after each discharge measurement, the meter will be examined for damage, wear, or faulty alignment. During measurements, the meter will be observed periodically when it is out of the water to be sure that the sensor is free of debris.

Meters will be cleaned daily when in use. If measurements are made in sediment-laden water, the meter will be cleaned immediately after each measurement.

#### 3.4.5 Field Procedures

Upon arrival at the site, field personnel will evaluate the flow conditions to determine which measurement method is appropriate. Based on flow conditions, the appropriate flow meter (e.g., Marsh-McBirney Model 2000 Flo-Mate meter, Hach FH950, or Son-Tek) will be selected to perform an area-velocity measurement.

At each measurement point (or section) across the stream crosssection, depth is measured prior to measurement of velocity. Place the wading rod about 5 feet downstream from the tagline. Place the wading rod in the stream so the base plate rests on the streambed. The depth of water is read from the graduated main rod. The main rod is graduated into 0.1-foot

increments. These increments are indicated by a single score in the metal. Half-foot increments are marked by two scores in the metal, and each foot is marked by three scores in the metal. A vernier scale on the upper handle of the rod corresponds to 0.1-foot increments, and has 1 through 9 in raised numbers next to raised marks. A sliding, adjustable rod, known as the setting rod, to which the meter is attached, has single scored marks that are aligned with values on the vernier scale.

In high-velocity areas, it is recommended that depth be read as the value between depth on the upstream side of the rod and depth on the downstream side of the rod. Depth is measured to the nearest 0.02 foot. This depth is used to set the vertical location of the current meter.

The setting rod is then adjusted downward so that the scored mark of the setting rod that corresponds to the range of depth in feet (e.g., if depth = 0.46, range in feet = 0; or if depth = 1.72, range in feet = 1) is aligned with the stream depth value transposed to the vernier scale. This automatically positions the meter for use in the 0.6 method as the meter is then six-tenths of the total depth from the surface of the water.

For using the two-point method of velocity measurement, the depth of water is divided by 2. This value is set so that the meter will be at the 0.8-depth position from the water surface. The depth of water is then multiplied by two, and this value is set. The meter will then be at the 0.2-depth position measured down from the water surface. These two positions represent the conventional 0.2- and 0.8-depth positions. If depths are less than 0.3 foot, the 0.5 method may be used. The observation depth recorded will then be 0.5 of the total depth.

If water quality or sediments are sampled in conjunction with discharge measurement, samples will be collected prior to making discharge measurements.

The following steps are to be followed in discharge measurement:

- ☐ Evaluate the measurement location. Choose a location where flow is least turbulent. If the prescribed location is in a stream reach with highly turbulent flow conditions, try to select a location immediately upstream or downstream. Flow should be visible from bank to bank. Eddies and slack water must not be present.
- ☐ Remove aquatic vegetation, ice, or other minor flow impediments. When such modifications are made, exercise great care to avoid unnecessary movement of sediments and allow flow to stabilize before the current meter measurement begins.
- ☐ Position a tape about 1 foot above the surface of the water. Secure the tape so that it remains taut and perpendicular to the channel.
- ☐ Select a starting point at either the left bank (left edge of water, LEW) or the right bank (right edge of water, REW). LEW and REW are determined when facing downstream.
- ☐ Note the distance in feet, and the stream direction, that this cross-section lies from the prescribed location. For example, the note may read "25 feet downstream" or "15 feet upstream."
- ☐ Measure the width of the stream. Select the number of subsections in which to measure velocity attempting to measure no more than 10 percent of the total flow in any one section, if possible.
- ☐ After determining the distance desired between measuring points, commonly referred to as sections, measurement can begin. Record the bank at which measurements start on the discharge measurement notes as "REW," using REW or LEW depending upon whether starting at the right or the left edge of the water.
- ☐ Note the distance to the beginning edge of water from the initial point. The initial point is an arbitrary point on the tape, preferably zero, which lies on the shoreline of the stream. All station locations are recorded as distances from the initial point.
- ☐ Proceed to the first station beyond the edge of water. Record the distance from the initial point on the discharge measurement notes. Place the wading rod into the stream so the base plate rests on the stream bed.
- ☐ Stand downstream of the tagline or tape and face upstream. Do not stand behind or close to the meter. Raise the current meter on the wading rod so that it is well above the surface of the water.



- ☐ Measure stream depth at the measurement point as indicated on the wading rod. Record the stream depth to the nearest 0.02 foot (for example 0.32 feet or 1.54 feet).
- ☐ Lower the meter to the required depth and record the observation depth. The observation depth as a fraction of total depth is 0.6, 0.2, 0.8 or occasionally 0.5.
- ☐ Field personnel will stand in a position that least affects the velocity of the water passing the current meter. That position is usually obtained by facing upstream with the arm fully extended. The person(s) will stand at about a 45-degree angle downstream from the wading rod. The wading rod is held in a vertical position with the meter parallel to the direction of flow. Avoid standing in the water when possible.
- ☐ Field personnel will wait for the velocity on the meter's screen to stabilize and record in the appropriate column. The flow meter must be aligned parallel to the direction of flow.
- ☐ Proceed to the next station. Record the distance from the initial point to the station. Repeat measurements of depth and velocity. Continue in this manner across the stream.
- ☐ After recording the distance measurement at the last station, record the ending edge of water that is reached (e.g., LEW [or REW]).
- ☐ Note velocity and depth at the edge of water as zero.
- ☐ Evaluate and record flow characteristics, weather conditions, air temperature, water temperature, observer(s), type of meter, and remarks.
- ☐ If an insufficient number of subsections have been used for the measurement, repeat the measurement steps. Begin from the opposite bank from where the previous measurement began.

#### 3.4.6 Discharge Calculations

- ☐ Calculate discharge on the discharge notes as follows: Use the distances from initial point to compute width for each subsection. The first width is computed by subtracting the first distance (edge of water) from the second distance and dividing this quantity by two. The second width will be the difference between the third distance and the first distance divided by two. For each subsequent width, subtract the previous station distance from the following station distance and divide this quantity by two. The final width is calculated as the difference between the final distance and the second-to-last distance divided by two. Sum the width column and check to ensure that the calculated width equals the distance between the REW and LEW.

- ☐ Multiply the width by the depth for each station to determine the area of each subsection. Sum the areas to determine total area.
- ☐ Multiply the velocity by the area for each station to obtain the discharge for each subsection.
- ☐ Sum the discharges for each subsection to determine total discharge and record the value.
- ☐ If two sets of discharge measurements beginning at opposite banks were taken, repeat the discharge calculations for the second set of data. Average the total discharges for the two measurements. Record the average value and report it for input into the database.

### 3.5 Control Structures

Control structures, such as flumes, can be used to determine discharge. These structures have regular dimensions that allow for a consistent relationship between water level and discharge. This section describes use of Parshall flumes to measure discharge.

#### 3.5.1 Introduction

A calibrated constriction placed in a stream channel changes the level of the water in or near the constriction. Flumes are constructed so that a restriction in the channel causes the water to accelerate, producing a corresponding change (drop) in the water level. When the physical dimensions of the flume constriction are known, discharge through constriction may be determined from measurement of depth. See below for a description of discharge measurement for Parshall flumes.

Typical flumes consist of three sections:

- ☐ A converging section to accelerate the approaching flow;
- ☐ A throat section, whose width is used to designate flume size; and
- ☐ A diverging section, designed to ensure that the level downstream is lower than the level in the converging section.

The stage of a stream is the height of the water surface above an established elevation. Stage is usually expressed in feet. The Parshall flume consists of a converging section with

a level floor, a throat section with a downward sloping floor and a diverging section with an upward sloping floor. The principal feature of the Parshall flume (developed by R. Parshall in 1922) is an approach reach having converging sidewalls and a level floor, the downstream end of which is a critical depth cross-section. The primary stage measurement is made in the approach reach at some standard distance upstream from the critical-depth cross-section.

The flumes are designated by the width (w) of the throat. Flumes having throat widths from 3 inches (in.) to 8 feet (ft.) have a rounded entrance whose floor slope is 25 percent. Smaller and larger flumes do not have that feature.

### 3.5.2 Required Measurement Conditions

Ideally, flow rate through a flume may be determined by measurements at a single point some distance downstream from the inlet and above the throat.

### 3.5.3 Equipment

The following equipment will be needed:

- ☐ Current meter
- ☐ Carpenter's level
- ☐ Framing square
- ☐ Measuring tapes
- ☐ Staff gauge

### 3.5.4 Maintenance and Calibration Procedures

All flumes will be inspected to determine that entrance conditions provide a uniform influent flow distribution, the converging throat section is level, and that the throat section walls are vertical. The flume will be closely examined to determine that it is discharging freely. Any problems observed during the inspection will be noted and reported to the field manager.

### 3.5.5 Procedures

Steps to be followed in measuring discharge.

- ☐ Remove any material that may have accumulated in the flume or on the weir;
- ☐ If the station includes a chart recorder, inspect the strip chart on the recorder to verify that it is operating;
- ☐ Note any deterioration of the station; report these conditions to the field manager at the conclusion of daily data collection activities;
- ☐ Measure and record the throat width (W) to the nearest 1/10 of an inch;
- ☐ Use the staff gauge to measure the gauge height (H) to the nearest 0.02 foot;
- ☐ Calculate discharge as described in Section 3.5.6; and
- ☐ Record the calculated discharge and the time and date of the site visit.

### 3.5.6 Discharge Calculations

A set of flume tables is necessary for calculating flows. The flume tables are specific to the type and size of flume and are usually supplied by the flume manufacturer. Based on the gauge height (head, H, in feet) and the throat width of the flume (size of flume, W), the discharge is read directly from the table provided from the manufacturer. Note that the approximate values of discharge for heads other than those shown may be found by direct interpolation in the table. The following equation and table gives calculation coefficients for discharge calculations with Parshall flumes.

The free-flow discharge equations for the standard Parshall flume sizes are of the form:

$$Q = Ch_a^n$$

where:

$h_a$  = measuring head (ft)  
 $Q$  = discharge (ft<sup>3</sup>/s)  
 $C$  and  $n$  = coefficients specific to flume size

Coefficients ( $C$ ) and exponents ( $n$ ) for Parshall flume discharge calculations are listed in the following table.

Throat width	Coefficient ( $C$ )	Exponent ( $n$ )
1 in	0.338	1.55
2 in	0.676	1.55
3 in	0.992	1.55
6 in	2.06	1.58
9 in	3.07	1.53
1 ft	3.95	1.55
2 ft	8.00	1.55
3 ft	12.00	1.57
4 ft	16.00	1.58
5 ft	20.00	1.59
6 ft	24.00	1.59
7 ft	28.00	1.60
8 ft	32.00	1.61
10 ft	39.38	1.60
12 ft	46.75	1.60
15 ft	57.81	1.60
20 ft	76.25	1.60
25 ft	94.69	1.60
30 ft	113.13	1.60
40 ft	150.00	1.60
50 ft	186.88	1.60

### 3.6 Documentation

Information required by this SOP will be documented in detail in a field notebook and/or on a Surface Water Flow Record form (attached form or similar available in paper or electronic format). This information includes the calibration data for flow measurement devices and field discharge measurement data.

Documentation will also include the type of flow measurement device, including a model number; a detailed description of measurement location and weather conditions during the measurement; and calculations.

## **4.0 QUALITY ASSURANCE/QUALITY CONTROL**

### **4.1 Calculation Check**

All calculations will be reviewed for accuracy and conformance with these procedures. The calculation review will be performed by a technically qualified individual before results are reported or interpreted. The calculation check shall be documented by recording the reviewer's initials and date of review on the calculation sheet. A copy of the reviewed calculations should be included in the project file.

### **4.2 Records Review and Management**

The Project Manager or designated reviewer will verify that documentation has been completed and filed per this procedure.

## **5.0 REFERENCES**

Rantz, S.E. et al., 1982. Measurement and Computation of Streamflow: Volume 1, Measurement of Stage and Discharge, U.S. Geological Survey Water Supply Paper 2175. U.S. Government Printing Office, Washington, D.C.

U.S. Department of the Interior Geological Survey (USGS), 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, U.S. Geological Survey, Reston, VA.

U.S. Environmental Protection Agency (USEPA), 1986. Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual. Environmental Services Division, Region IV, Athens, GA.

## **SOP No. 7 Equipment Decontamination**

## **STANDARD OPERATING PROCEDURE No. 7**

### **EQUIPMENT DECONTAMINATION**

#### **1.0 SCOPE AND APPLICABILITY**

This Standard Operating Procedure (SOP) describes the methods to be used for decontamination of all reusable field equipment that could become contaminated during use and/or sampling. Field equipment may include split spoons, reusable bailers, trowels, scissors, shovels, hand augers, or any other type of equipment used during field activities. Decontamination is performed as a quality assurance measure and a safety precaution; it prevents cross contamination between samples and also helps to maintain a clean working environment. The procedures presented herein are intended to be general in nature and are applicable when referenced by site-specific or project-specific planning documents. Appropriate revisions may be made to accommodate site-specific conditions or project-specific protocols when approved in writing or via email by the Project Manager.

Decontamination is achieved primarily by rinsing with liquids which may include: soap and/or detergent solutions, potable water, distilled weak acid solution, and/or methanol or other solvent. Equipment may be allowed to air dry after being cleaned or may be wiped dry with chemical-free towels or paper towels if immediate re-use is necessary.

At most project sites, decontamination of equipment that is re-used between sampling locations will be accomplished between each sample collection point. Waste produced by decontamination procedures, including waste liquids, solids, rags, gloves, etc., should be collected and disposed of properly, based upon the nature of contamination. Specific details for the handling of decontamination wastes are addressed in SOP No. 3 (STORAGE AND DISPOSAL OF SOIL, DRILLING FLUIDS, AND WATER GENERATED DURING FIELD WORK) or may be specified by a project plan.



## **2.0 BASIS FOR METHODOLOGY**

The methods and procedures described in this SOP were developed from these sources:

- ASTM D5088. Standard Practice for Decontamination of Field Equipment Used at Waste Sites. American Society for Testing and Materials (ASTM) International, West Conshohocken, PA, 2008.
- Parker and Ranney, 1997a. Decontaminating Ground Water Sampling Devices, CRREL Special Report 97-25, U.S. Army Engineer Cold Regions Research and Engineering Laboratory, Hanover, NH.
- Parker and Ranney, 1997b. Decontaminating Materials Used in Ground Water Sampling Devices, CRREL Special Report 97-24, U.S. Army Engineer Cold Regions Research and Engineering Laboratory, Hanover, NH.

## **3.0 PROCEDURES**

### **3.1 Responsibilities**

It is the responsibility of the field sampling supervisor to ensure that proper decontamination procedures are followed and that all waste materials produced by decontamination are properly managed. It is the responsibility of the project safety officer to draft and enforce safety measures that provide the best protection for all persons involved directly with sampling and/or decontamination.

It is the responsibility of any subcontractors (e.g., drilling contractors) to follow the proper, designated decontamination procedures that are stated in their contracts and outlined in the Site-Specific Health and Safety Plan. It is the responsibility of all personnel involved with sample collection or decontamination to maintain a clean working environment and ensure that any contaminants are not negligently introduced to the environment.

### 3.2 Supporting Materials

Materials needed for equipment decontamination include:

- ☐ Cleaning liquids: laboratory grade soap and/or detergent solutions (Alconox, etc.), potable water, distilled water, methanol, weak nitric acid solution, etc.
- ☐ Personal protective safety gear as defined in the Site-Specific Health and Safety Plan
- ☐ Chemical-free towels or paper towels
- ☐ Disposable nitrile gloves
- ☐ Waste storage containers: drums, boxes, plastic bags, etc.
- ☐ Cleaning containers: plastic and/or stainless steel pans and buckets
- ☐ Cleaning brushes
- ☐ Aluminum foil

### 3.3 Methods

The extent of known contamination will determine the degree of decontamination required. If the extent of contamination cannot be readily determined, cleaning should be done according to the assumption that the equipment is highly contaminated. Decontamination procedures should account for the types of contaminants known or suspected to be present. In general, high levels of organic contaminants should include an organic solvent wash step, and high levels of metals contamination should include a weak acid rinse step.

The procedures listed below constitute the full field decontamination procedure. If different or more elaborate procedures are required for a specific project, they may be specified in the project planning documents. Such variations in decontamination protocols may include all, part, or an expanded scope of the decontamination procedure stated herein.

1. Remove gross contamination from the equipment by dry brushing, and rinse with potable water.
2. Wash with laboratory-grade detergent solution.

3. Rinse with potable water.
4. Rinse with methanol (optional, for equipment potentially contaminated by organic compounds).
5. Rinse with acid solution (optional, for equipment potentially contaminated by metals).
6. Rinse with distilled or deionized water.
7. Repeat entire procedure or any parts of the procedure as necessary.
8. Air dry.

#### **4.0 DOCUMENTATION**

Field notes will be kept describing the decontamination procedures followed. The field notes will be recorded according to procedures described in SOP No. 1 (FIELD DOCUMENTATION).

#### **5.0 QUALITY CONTROL**

To assess the adequacy of decontamination procedures, field rinsate blanks may be required. The specific number of field rinsate blanks will be defined in the project-specific Sampling and Analysis Plan (SAP) or Quality Assurance Project Plan (QAPP).

Rinsate blanks with elevated or detected contaminants will be evaluated by the Project Manager, who will relay the results to the field personnel. Such results may be indicative of inadequate decontamination procedures that require corrective actions (e.g., retraining).

#### **6.0 REFERENCES**

ASTM D5088-02 (2008). Standard Practice for Decontamination of Field Equipment Used at Waste Sites. American Society for Testing and Materials (ASTM) International, West Conshohocken, PA, 2008. Available online at <http://www.astm.org/>

Parker and Ranney, 1997a. Decontaminating Ground Water Sampling Devices, CRREL Special Report 97-25, U.S. Army Engineer Cold Regions Research and Engineering Laboratory, Hanover, NH.

Parker and Ranney, 1997b. Decontaminating Materials Used in Ground Water Sampling Devices, CRREL Special Report 97-24, U.S. Army Engineer Cold Regions Research and Engineering Laboratory, Hanover, NH.

## **SOP No. 20 Data Review and Validation**

## **STANDARD OPERATING PROCEDURE No. 20**

### **DATA REVIEW AND VALIDATION**

#### **1.0 SCOPE AND APPLICABILITY**

This Standard Operating Procedure (SOP) describes the procedures for the evaluation of data generated through inorganic laboratory analysis of samples. These procedures apply to three levels of data evaluation: data completeness check, data review and data validation.

The QAPP, Sampling and Analysis Plan (SAP) and/or any other relevant site-specific or project-specific documents must be reviewed before this SOP is used to evaluate data. The individual performing the data evaluation shall be familiar with the analytical methods and other procedures used for the project. Familiarity with project and laboratory quality control requirements is critical to appropriate use of this procedure. A general description of the different levels of data evaluation is provided below and discussed in detail in Section 4.0 of this SOP.

#### **1.1 Data Completeness Check**

Data completeness checks may be performed on both Level 2 standard data reports and Level 4 USEPA Contract Laboratory Program (CLP)-like laboratory reports as specified in the project planning documents and/or by the project team or regulatory agencies.

These completeness checks may be performed as part of a data review or validation or may be performed as a stand-alone evaluation. Completeness checks only document the presence or absence of applicable QC data in the laboratory data package, and no qualification of sample results is necessary based on this data evaluation.

#### **1.2 Data Review**

Data review includes a review of laboratory quality assurance (QA) and quality control (QC) sample results provided in Level 2, or equivalent, standard laboratory reports. Data review can also be performed on CLP-like Level 4 data packages if required. In addition to sample results, Level 2 laboratory reports provide QA/QC summaries that

typically include results for method blanks, laboratory control samples (LCS), matrix spike (MS) samples, and duplicates, as well as the review of field QC samples (e.g., field blanks and field duplicates). Data review is differentiated from data validation because the review consists of an assessment of the laboratory QA/QC summary reports only.

### 1.3 Data Validation

Data validation includes the evaluation of the QA/QC results described above as well as an evaluation of additional validation of calculations, calibrations, internal standards, tunes, etc. provided in Level 4 CLP-like data reports. A minimum of 10% of the data reports produced annually by each laboratory analyzing environmental monitoring samples will be reported as CLP-like data reports and validated according to the data validation procedures described in this SOP (Section 4.3). Data validation of the CLP-like data reports will be performed using the general protocols and processes described in this SOP, as applicable to the method calibration and QC limits specified on Tables 2-2 through 2-6 of the QAPP, the Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (NFG; USEPA, 2010) and to the extent possible when certain non-CLP methods are used, laboratory SOPs.

The following table summarizes the common elements and differences between a data completeness check, data review and data validation.

**Scope of Data Reviews**

Item	Data Completeness Check	Data Review	Data Validation
Review of Work Plan, SAP and/or QAPP	Presence only	X	X
Review of Chain-of-Custody Records	Presence only	X	X
Review of Case Narrative	Presence only	X	X
Verify that preservation and holding time requirements met.	Presence only	X	X
Verify that the required frequency of field QC samples was met.	Presence only	X	X
Verify that ICP/MS tune analyses were performed at the required frequency and that results are within control limits.			X

Verify that all instrument calibration were performed at the required frequency and concentrations and that results are within control limits.			X
Verify that laboratory blanks were performed at the required frequency and that results are within the control limits.	Presence only	X	X
Verify that field blank results are within the control limits.	Presence only	X	X
Verify that all Laboratory Control Sample (LCS) were performed at the required frequency and that results are within control limits.	Presence only	X	X
Verify that matrix spike (MS) sample were performed at the required frequency and that results are within control limits.	Presence only	X	X
Verify that analytical duplicates were performed at the required frequency and that RPDs are within control limits.	Presence only	X	X
Verify that ICP Serial Dilutions were performed at the required frequency and that results are within control limits.			X
Verify that ICP/MS internal standards were included with each sample and that results are within control limits.			X
Verify that field duplicate measurements are within the control limits.	Presence only	X	X
Verify sample calculations.			X
Verify that project completeness goals were met.		X	X

## 2.0 BASIS FOR METHODOLOGY

The data evaluation procedures described in this SOP are based on the guidance specified in the QAPP and the protocols specified in the USEPA Contract Laboratory Program (CLP) National Functional Guidelines (NFGs) for Inorganic Superfund Data Review (USEPA, 2010). The data evaluation procedure described in this SOP may be used for the evaluation of standard laboratory data reports (Level 2 reports) or CLP-like/Level 4 laboratory data reports. CLP-like/Level 4 data reports are needed in order to complete the validation procedure described in this SOP. It is not meant to replace or incorporate all of the procedures and protocols necessary to complete data validation



per the USEPA NFGs. Data qualification may or may not be performed for data review, however data validation will include data qualification.

### 3.0 DEFINITIONS

Definitions of accuracy, precision, and completeness and methods for computing their measures are provided below. Descriptions of the contents of Level 2 Standard data packages and Level 4 CLP-like data packages are provided in Section 4.2 of this SOP.

#### a. Accuracy

Accuracy is the degree of difference between the measured or calculated value and the true value. Data accuracy and analytical bias are often evaluated by the analysis of LCS and MS samples, with results expressed as a percentage recovery measured relative to the true (known) concentration.

The percentage recovery for LCS samples is given by:

$$\text{Recovery (\%)} = \frac{A}{T} \times 100$$

where: A = measured concentration of the surrogate or LCS; and  
T = known concentration.

The percentage recovery for MS samples is given by:

$$\text{Recovery (\%)} = \frac{A - B}{T} \times 100$$

where: A = measured concentration of the spiked sample;  
B = concentration of unspiked sample; and  
T = amount of spike added.

Laboratory blanks, and often, field blanks are analyzed to quantify artifacts introduced during sampling, transport, or analysis that may affect the accuracy of the data.

#### **b. Precision**

Precision is the level of agreement between duplicate measurements of the same characteristic. Laboratory precision, or analytical error, is assessed by determining the agreement of results for replicate measurements of the same sample. Field precision is assessed by determining the agreement for results for two independent samples collected from the same site at the same time. Precision may be evaluated using LCS/LCSD samples, MS/MSD samples, analytical duplicate samples and/or field duplicate samples. The comparison is made by calculating the relative percent difference (RPD) as given by:

$$\text{RPD (\%)} = \left| \frac{2(S1 - S2)}{S1 + S2} \right| \times 100$$

where: S1 = measured sample concentration; and  
S2 = measured duplicate concentration.

#### **c. Completeness**

Completeness is the percentage of usable data measurements obtained, as a proportion of the number of data measurements planned for the project. Completeness is affected by such factors as sample bottle breakage and acceptance/non-acceptance of analytical results. Percentage completeness (C) is given by:

$$C (\%) = \frac{V}{P} \times 100$$

where: V = number of usable data measurements obtained; and  
P = number of data measurements planned.

#### d. Data Qualifier Flags

As a result of the data review or validation procedures (but not data completeness checks), data qualifier flags may be applied to individual analytical results if qualification for project data usability is appropriate. Definitions of the flags applied for data qualification are as follows:

<u>Flag</u>	<u>Definition</u>
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

An explanation regarding the assignment of qualifiers in accordance with the review procedures is detailed below in Section 4.2.

## 4.0 PROCEDURES

The data evaluation documentation requirements and procedures for data completeness checks, data review, and data validation are described below in the following sections.

#### 4.1 Data Completeness Check Procedure

Data completeness checks can be performed as a stand-alone evaluation or as part of a full data review or validation. A data completeness check is performed to verify that the laboratory data provided are complete. The following shall be reviewed for Level 2 Standard data reports and Level 4 CLP-like data reports.

Level 2 Standard data reports shall include the following information for each sample:

- ☐ Field and laboratory sample identification;
- ☐ Sample result, method detection limit, and reporting limit, with appropriate units;
- ☐ Dilution factor
- ☐ Sample collection, receipt, and analysis dates;
- ☐ Analytical method(s) references; and
- ☐ Laboratory qualifiers and definitions.

In addition, Level 2 Standard data reports shall include the following information in a QA/QC summary:

- ☐ Method blank results for each analyte;
- ☐ LCS results and laboratory control limits for each analyte;
- ☐ MS results and laboratory control limits for each analyte, if applicable;
- ☐ Analytical duplicate results and laboratory control limits for each target analyte (LCSD and/or MSD results may be provided instead of analytical duplicate results); and
- ☐ Confirmation of instrument calibration; and
- ☐ Copies of the signed COCs.

Level 4 CLP-like laboratory reports shall include the following information for each sample, at a minimum:

- ☐ Field and laboratory sample identification;
- ☐ Sample result, method detection limit, and reporting limit, with appropriate units;
- ☐ Sample collection and receipt dates;
- ☐ Sample preparation and analysis date/time;
- ☐ Dilution factor;
- ☐ Preparation and analysis batch numbers or identification;

- ☐ Sample matrix;
- ☐ Analytical method(s) references;
- ☐ Percent moisture determination; and
- ☐ For solid-matrix samples, identify basis of reporting (i.e., wet-weight or dry-weight basis).

The following additional information will also be provided in Level 4 CLP-like data reports, as applicable for the reported analytical methods:

- ☐ Case narrative;
- ☐ Copies of the signed COCs;
- ☐ Laboratory method/preparation blank;
- ☐ Initial calibration verification (ICV), and continuing calibration verification (CCV);
- ☐ Initial calibration blanks (ICB), and continuing calibration blank (CCB);
- ☐ Interference check sample, if applicable;
- ☐ Matrix spike (MS), and when applicable matrix spike duplicate (MSD), sample recovery and, when applicable, MS/MSD relative percent difference (RPD);
- ☐ Post-digest spike sample recovery;
- ☐ Laboratory duplicate;
- ☐ Laboratory control sample (LCS) recovery;
- ☐ ICP and ICPMS serial dilution percent differences;
- ☐ MDLs;
- ☐ ICP inter-element correction factors;
- ☐ ICP and ICPMS linear ranges;
- ☐ Preparation log;
- ☐ Analysis run log;
- ☐ Instrument raw data for verification;
- ☐ ICPMS tunes;
- ☐ ICPMS internal standards relative intensity summary;
- ☐ Sample log-in sheet; and
- ☐ Deliverables inventory sheet.

## 4.2 Data Review Procedure

The data review procedure for review of a Level 2 Standard data report is as follows. Data may or may not be qualified during data review depending on the project specifications.

- A. Review copies of the Chain-of-Custody records (COCs). Verify that all necessary information was provided on each COC and that all necessary signatures are present. Review laboratory records of sample temperature upon receipt and preservation information, if available, to verify that samples were properly preserved. Professional judgment may be used to determine if data qualification is necessary due to temperature exceedances and/or preservation deviations. Verify that all samples listed on the COCs were analyzed for the appropriate parameters. Note any problems documented on the COCs by either the laboratory or the sampler.
- B. Briefly review and summarize the laboratory case narrative, if present. Note any data that are indicated as outside of control limits.
- C. For each sample and each parameter, verify that the analyses were performed within the recommended holding time. For sample analyses performed outside the recommended holding times, sample results may be qualified as described in the QAPP or USEPA NFGs (2010), though professional judgment and project-specified requirements should be used.
- D. Identify any field QC samples and verify that the field QC samples specified in the Work Plan, QAPP or other relevant project documents have been collected at the correct frequency.
- E. Review the results of all field/equipment blanks and the laboratory method blanks. If an analyte was detected in a blank, the corresponding sample concentrations will be compared to the blank concentrations. Sample results may be qualified as described in the QAPP or USEPA NFGs (2010), though

professional judgment should be used to carefully evaluate the effect of blank concentrations on the sample data.

- F. Check the matrices, units, detection limits and reporting limits to verify that they are reported correctly and meet the project-specific requirements, if provided.
- G. Review all LCS (and LCSD, if available) recoveries and verify that they were within the project-specified control limits. If project-specific control limits are not provided, use the laboratory's control limits. LCS materials may not be available for all matrices. Sample results may be qualified as described in the QAPP or USEPA NFGs (2010), though professional judgment and project-specified requirements should be used.
- H. Review all MS (and MSD, if available) recoveries and verify that they were within the project-specified control limits. If project-specific control limits are not provided, use the laboratory's control limits. If analyzed and reported, post-digestion spike information should also be reviewed. Sample results may be qualified as described in the QAPP or USEPA NFGs (2010), though professional judgment and project-specified requirements should be used. For MS results that do not meet the control limits, the reviewer may choose to apply qualifiers to all samples of the same matrix associated with the MS, if the reviewer considers the samples sufficiently similar.

If an analytical duplicate was analyzed, compare the laboratory calculated RPD and compare this to the project-specified control limits. If a project-specific control limit is not available, use the laboratory's control limits. However, if one or both of the results are less than five times the PQL, use  $\frac{1}{2}$  PQL as the control limit for aqueous samples and  $2 \times \frac{1}{2}$  PQL as the control limit for non-aqueous (i.e., soil, sediment, tissue) sample matrices unless project-specific control limits are provided. If the analytical duplicate results fall outside of the control limits, sample results may be qualified as described in the QAPP or USEPA NFGs (2010), though professional judgment and

project-specified requirements should be used, LCS/LCSDs and/or MS/MSDs may be analyzed in place of, or in addition to, an analytical duplicate. The RPDs for LCS/LCSD and MS/MSD pairs shall be evaluated in the same manner as described above for analytical duplicates.

- I. If field duplicates were analyzed, calculate the RPD for each parameter and compare the RPDs to project-specified control limits. If project-specific control limits are not available, use 30 percent for aqueous samples and 50 percent for soil/solid/vegetation tissue samples. However, if one or both of the results are less than five times the PQL, use  $\frac{1}{5}$  PQL as the control limit for aqueous samples and  $2 \times \frac{1}{5}$  PQL as the control limit for non-aqueous (i.e., soil, sediment, tissue) sample matrices unless project-specific control limits are provided. If the field duplicate results fall outside of the control limits, the associated field duplicate results should be qualified in the same manner described above for analytical duplicates as described in the QAPP or USEPA NFGs (2010), though professional judgment and project-specified requirements should be used. Professional judgment will be used to determine whether additional sample results, in addition to the field duplicate sample results, should also be qualified.
- J. Determine whether the project's analytical completeness goal was met. Note any rejected data.

The data reviewer may also provide a brief summary of the accuracy, precision and completeness of the data set. The qualifier flags assigned to the data will be summarized in a table and/or entered into the electronic data deliverable, as specified in the project's QAPP or SAP.

#### **4.3 Data Validation Procedure**

A minimum of 10% of the data reports produced annually by each laboratory analyzing environmental monitoring samples from Smoky Canyon Mine will be reported as CLP-like data reports and validated according to the data validation procedures described in this SOP. The data validation procedure shall include all of the above steps in the data



review procedure with additional steps as outlined below. These additional steps include the recalculation of instrument and sample results from the laboratory instrument responses for a subset of the data. These recalculated results are compared to the laboratory reported results to confirm that the instrument outputs were correctly reported. Also, additional QC summary reports will be reviewed including the ICP/MS tune summary, the instrument calibrations, the interference check sample summary, the serial dilution sample summary, and the internal standard relative intensity summary. Data will be qualified during the data validation procedure with the appropriate qualifiers as specified in the QAPP and consistent with USEPA's NFG (2010). A more complete description of the additional steps to be followed in data validation is presented below.

- A. Verify sample calculations for a few of each sample results and identify and document any calculation errors if any are present. The raw instrument output will be reviewed to confirm that the analyte concentrations were reported correctly.
- B. Verify that the ICP/MS tune analysis data requirements were met and results were within QC limits. Review the raw data for a subset of the tune results and confirm that the raw data matches the results summarized on the ICP-MS Tune summary form. If the ICP/MS tune analysis results fall outside of the control limits, the associated sample results should be qualified as described in the QAPP or USEPA NFGs (2010).
- C. Verify that the instrument calibration was performed at the required frequency, that results are within QC limits, and review associated standards, including initial and continuing calibration standards and blanks. For a subset of the analytes, recalculate the percent recoveries for calibration standards using the data on the Initial and continuing calibration verification summary form and verify that the concentrations reported on this form are consistent with those in the instrument output. For ICVs/CCVs that have percent recoveries outside of control limits and for calibration blanks for which analytes are detected, review the run logs to confirm which samples were affected by out of control CCVs and CCBs. Associated sample results should be qualified as described in the QAPP or USEPA NFGs (2010) though professional judgment and project-specified requirements should be used.

- D. Verify that Interference Check Sample data requirements were met and results are within QC limits. Recalculate a subset of the percent recoveries and review the raw data to verify that the results from the instrument output match those reported on the Interference Check Sample summary form. If the interference check sample results fall outside of the control limits, the associated sample results should be qualified as described in the QAPP or USEPA NFGs (2010).
- E. Verify that ICP serial dilutions requirements were met and results are within QC limits. Recalculate percent differences for a subset of the results and verify that instrument outputs match values reported in the summary form. Where percent differences exceed the control limit and sample results are greater than 50 times the method detection limit, the associated sample results should be qualified as described in the QAPP or USEPA NFGs (2010).
- F. Verify that ICP/MS internal standard requirements were met and results within QC limits. Review raw data and recalculate a subset of the relative intensities of the internal standards and compare them to those reported on the internal standard relative intensity summary form. The associated sample results should be qualified as described in the QAPP or USEPA NFGs (2010).

Qualify all sample data associated with QC or calibration that do not meet the project specifications or QC limit using the appropriate qualifiers as defined in Section 3.4 Data Qualifiers. Use the guidance for data qualification from the project specific guidelines in the QAPP or guidance in the USEPA NFG (2010).

## 5.0 DOCUMENTATION

The data evaluation procedures and results will be documented through completion of a checklist, worksheet or summary document, subject to review and approval by the appropriate project representative(s). The data evaluation documents will be provided to the Project Manager and included in the project file containing the associated laboratory result reports.

Include the project name, project number, laboratory name, laboratory project number, field sample IDs, sample matrix, and analytical parameters and methods used on the data evaluation documentation forms. Specify the relevant project-planning documents and reference the protocol that was used to perform the data evaluation (such as this SOP).

A data review checklist is provided in Attachment A and a data validation checklist is provided in Attachment B. The table in Section 1.3 or list of report contents in Section 4.1 can be used as the basis for a checklist of the data completeness check.

## 6.0 DATA USE

Qualifier flags are assigned to describe the degree to which individual values provide accurate and precise results. The general criteria for assigning flags and their meaning in terms of future data use are as follows:

- ☐ Values assigned J flags (J, J+, or J-) are considered estimated results. QC data supplied with those values indicate that they may not be accurate or precise within the limits specified in the QAPP or a project-specific document, but that the magnitude of the potential imprecision or inaccuracy is not great enough to reject the value for project data uses.
- ☐ Values assigned R flags do not meet the requirements for accuracy, precision, representativeness, or reproducibility specified to provide quantitative data for the project data uses. The R flag indicates that serious deficiencies were encountered preventing the generation of usable data for the project objectives.
- ☐ Values assigned U flags indicate that a concentration of the analyte cannot be confirmed due to the presence of an interferant or the presence of the analyte in associated blanks. UJ flags may be applied to indicate that the presence of the analyte cannot be confirmed and the value of the reported quantitation limit for the sample may not be accurate or precise. Values flagged with U or UJ are fully usable and should be considered undetected.
- ☐ Values without flags assigned have met all of the project data quality objectives and are suitable for all project data uses.

## 7.0 QUALITY ASSURANCE/QUALITY CONTROL

The data evaluation documents will be reviewed internally for conformance to the procedures described herein. Once any questions or comments resulting from that

review have been resolved, the data evaluation documents will be considered final and any data qualifiers will be assigned to the results that are ultimately included in the project's electronic database.

## **8.0 REFERENCES**

U.S. Environmental Protection Agency (USEPA), 2010. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review. EPA 540-R-10-011. January.

## **SOP No. 31 Water Quality Meter Calibration**

## **STANDARD OPERATING PROCEDURE No. 31**

### **WATER QUALITY METER CALIBRATION**

#### **1.0 SCOPE AND APPLICABILITY**

This Standard Operating Procedure (SOP) describes the protocol to be followed for calibration of the field water quality sampling multi-parameter instrument used during environmental monitoring and sampling activities. The procedures presented herein are intended to be general in nature and are applicable when referenced by site-specific or project-specific planning documents. Appropriate modifications to the procedures may be made to accommodate project-specific protocols when approved in writing or via email by the Project Manager or detailed in a project work plan, sampling plan, or quality assurance project plan.

The objective of calibrating field instruments is to establish the accuracy and reliability of the instrument and to ensure that field readings are consistent with other measurements.

#### **2.0 BASIS FOR METHODOLOGY**

The methods and procedures described in this SOP were developed from this source:

- U.S. Geological Survey (USGS) (variously dated) National Field Manual for the Collection of Water-Quality Data: U.S. Geological Survey Techniques of Water-Resources Investigations, Book 9, Handbooks for Water-Resources Investigations, Chapters A1-A9.

### **3.0 EQUIPMENT AND SUPPLIES**

#### **3.1 Multi-Parameter Sensors**

Multi-parameter sensors can vary between manufacturers of instruments and as technology advances. The following are the sensors generally used on multi-parameter instruments for collecting water quality parameters: water temperature, pH, specific electrical conductance (SC), oxidation-reduction potential (ORP), and dissolved oxygen (DO). Turbidity is generally measured using a separate meter, but there are some instruments for which the turbidity sensor is included with the multi-meter sensor cluster.

- ☐ pH – sensor has a range between 2 to 12, or 0 to 14 pH units
- ☐ Water Temperature – sensor has a range of at least -5 to +45 degrees Celsius
- ☐ SC – sensor is temperature compensating, and measures in micro siemens per centimeter (uS/cm) or mS/cm
- ☐ DO – 2 types of sensors (polarographic and optical) both sensors range from 0.05 to 20 milligrams per liter (mg/L)
- ☐ ORP – sensor uses a platinum electrode, and measures in millivolts (mV)

#### **3.2 Calibration Supplies**

The following supplies are needed to calibrate a multi-parameter instrument: specific sensor buffers, standards, and calibration solutions, field notebooks, deionized water, bucket(s), disposable gloves, scrub brushes, and paper tissues.

### **4.0 CALIBRATION PROCEDURE**

The multi-parameter instrument will be calibrated in the field once daily by personnel according to manufacturer's instructions prior to the collection of any samples. All calibration details will be recorded in a field notebook including, but not limited to: instrument type, instrument serial number, readings prior to calibration, buffers used, readings after calibration, names of personnel calibrating, and date and time of calibration. The following are general guidelines to follow when calibrating a multi-parameter instrument:

- A. Follow the manufacturer instructions;
- B. Set the meter to the correct measurement units;
- C. Allow the meter to warm up (at least 10 minutes or according to manufacturer recommendation);
- D. Calibrate the instrument in a temperature-stable environment;
- E. Use the calibration cup for calibration;
- F. Use the recommended volume of calibration solution during calibration;
- G. Do not over tighten the calibration cup;
- H. Rinse the sensor with deionized water prior to the use of calibration solution, then rinse with a small amount of the calibration solution to be used before calibrating; and
- I. Calibrate the meter sensors in the following order: water temperature, SC, DO, pH, and ORP.

#### **4.1 Multi-Point Calibration**

##### **4.1.1 Water Temperature**

Check to ensure the accuracy of the temperature sensor at least every 3 months if the multi-parameter instrument is in frequent use or according to the manufacturer's recommendations. The accuracy of the temperature sensor will be verified against a certified NIST-traceable digital or liquid-in-glass thermometer. Completely submerge the multi-parameter meter temperature sensor and allow at least 1 minute for the temperature to equilibrate and stabilize. Record the temperature value in degrees Celsius (°C). If the difference between the readings does not fall within the manufacturer-specified accuracy, contact the supplier or manufacturer for the next steps.

##### **4.1.2 Specific Conductance (SC)**

Calibration for SC is performed using a one-point calibration. Use the standard recommended by the manufacturer or a standard that is similar in conductivity to the sample water. The calibration cup and sensor will first be rinsed using a small amount of calibration solution prior to the start of calibration. Next the calibration cup will be filled with the recommended volume of calibration solution and the sensor completely



submerged. When the readings stabilize save the calibration point and record in the field notebook the readings before and after calibration in uS/cm.

#### 4.1.3 Dissolved Oxygen (DO)

Follow the manufacturer's guidelines for care, proper setup, and calibration of the DO sensor for the instrument in use. Whenever possible, ensure that the DO sensor has been appropriately calibrated by the instrument supplier or party responsible for maintenance prior to using the instrument in the field.

#### 4.1.4 pH

Calibration of the pH sensor is performed using a two-point calibration. Select the pH 7 buffer as well as a second pH buffer (pH 4 or pH 10) that brackets the expected range of sample water pH. A calibration check using a third buffer can be performed at the end of calibration. To start, the calibration cup and sensor will be rinsed with deionized water and then with a small amount of the first buffer. Next the calibration cup will be filled with enough of the first buffer to completely cover the pH and temperature sensors (the pH value is temperature dependent). Wait for the pH and temperature sensors to equilibrate to the temperature of the buffer and record the temperature reading after stabilization. Adjust the calibration reading (to the true pH value at that temperature) using the chart provided by the buffer manufacturer. Record the temperature and pH readings before and after calibration of the first buffer in the field notebook. Follow the same steps starting with the rinsing of the calibration cup and sensor for the second buffer. If a third buffer is used to check the calibration, follow the same steps, but do not lock in a calibration point.

#### 4.1.5 Oxidation-Reduction Potential (ORP)

Calibration of the ORP sensor is generally performed using a one-point calibration at a known temperature. The manufacturer's recommendation will be followed for calibration. The calibration cup and sensor will first be rinsed with a small amount of the solution. Next fill the calibration cup with enough of the solution to completely submerge the ORP sensor. Wait for the readings to stabilize and then enter the correct value of

the solution at the current temperature. Record the ORP readings before and after calibration in mV, as well as the solution values used in the field notebook.

## 4.2 One-Point Calibration

Calibration may be performed using the In-Situ Quick Cal Solution when an In-Situ smarTROLL™ MP handheld water quality meter is used. The Quick Cal Solution performs a one-point calibration of all smarTroll™ MP sensors (pH, ORP, SC, and DO) at the same time. The manufacturer's recommendations will be followed for calibration as well as the following use and storage guidelines:

- ☐ Shake well before use;
- ☐ Allow to warm to room temperature before using;
- ☐ Store in refrigerator (needs to be stored in dark and cool environment);
- ☐ Use within three weeks after opening (document on bottle and calibration records when opened);
- ☐ Unopened shelf life is six months (check and document expiration date of bottle); and
- ☐ One-time use only (i.e. solution should not be re-used following single calibration).

## 5.0 DOCUMENTATION

The Project Manager or designated reviewer will check and verify that documentation of instrument calibration has been completed and the calibration records are filed in the project records.

## 6.0 REFERENCES

U.S. Geological Survey (USGS), variously dated. National Field Manual for the Collection of Water-Quality Data: U.S. Geological Survey Techniques of Water-Resources Investigations, Book 9, Handbooks for Water-Resources Investigations, Chapters A1-A9. Available online at <http://pubs.water.usgs.gov/twri9A>.


# **EPA SOP FLD#-5 Standard Operating Procedure for Soil Sampling**

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Replaces SOP: N/A

### Soil Sampling

#### APPROVED:

  
ESAT Region 8 QA Coordinator

Date 07/16/13

  
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Date 7/16/13

  
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Date 7/16/2013

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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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Appendix A: Soil Sampling Equipment

Appendix B: Auger and Trier Diagrams

Appendix C: Hand Auger Operating Instructions

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## 1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a guideline for the collection of representative soil samples in the field. The collection and analysis of soil samples serves to establish whether pollutants are present in the soils and helps determine the required action level(s) with regard to public and environmental health and welfare.

## 2.0 SCOPE AND APPLICATION

These are standard operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and included with the final report. Mention of trade names or commercial products does not constitute Techlaw, inc. endorsement or recommendation for use.

## 3.0 SUMMARY OF METHOD

Soil samples may be collected using a variety of methods and equipment. The methods and equipment used are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or a backhoe if necessary.

## 4.0 ACRONYMS AND DEFINITIONS

COC	Chain of Custody
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
OSHA	Occupation Safety and Health Administration
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team

Chain of Custody (COC) – A chronological document that tracks transfer of samples between entities from collection to disposal

Composite Sampling – Sampling from several points or intervals and consolidating them into a larger sample

Discrete Sampling - Sampling from a single location

Health and Safety Plan (HASP) – A site-specific plan that outlines potential hazards and procedural/equipment recommendations

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Standard Operating Procedure (SOP) - A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007)

## **5.0 HEALTH AND SAFETY**

When working with potentially hazardous materials or in hazardous situations, personnel must understand and comply with the site-specific Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP) and Health and Safety Plan (HASP) before the sampling event begins. More specifically, when sampling waste rock piles or fluvial deposit zones containing known or suspected hazardous substances, adequate personal protective equipment such as nitrile gloves, safety glasses, and protective footwear are necessary to prevent exposure.

When traversing tailings piles, hazardous situations exist that require the sampling personnel to wear adequate safety equipment including gloves and non-slip footwear. Never perform sampling activities if it cannot be done so in a safe manner (tailings piles are too steep, lightning is occurring, etc).

## **6.0 CAUTIONS**

There are cautions to be considered before deployment on a soil sampling event. If the samples are to be collected in an urban area at depth, the underground utility lines must be identified. In addition, if sampling at a remote waste rock pile, always use the buddy system when traversing steep gradients that may present fall hazards. Always review the site-specific HASP for potential safety hazards.

## **7.0 INTERFERENCES**

There are two primary interferences or potential problems associated with soil sampling. These include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve disturbance of the matrix (i.e. walking on specific areas that will ultimately be sampled) resulting in compaction of the sample or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

## **8.0 PERSONNEL QUALIFICATIONS**

Any personnel involved with field sampling activities must be cleared for health and safety. Clearance includes medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. Personnel who will be collecting soil samples should familiarize themselves with this and other pertinent SOPs such as the Sample Equipment Decontamination SOP FLD 02.00, the Sample Preservation SOP FLD 03.00, the Sample Custody and Labeling SOP FLD 11.00, and the General Field Sampling Protocols SOP FLD 12.00.

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## 9.0 EQUIPMENT

Equipment needed for collection of soil samples may include:

HASP Gear - Gloves, proper footwear, safety glasses, etc.

Mapping and Location Tools – Global Positioning System (GPS) units, site/local area maps, compass, tape measure, survey stakes, pin flags, camera, 2-way radios

Documentation Tools – Field log book, field data sheet, COC(s), labels, clear tape, pens, permanent marker, waterproof paper

Sampling Tools – Plastic, Teflon™, or other appropriate composition scoop (analysis dependent), shovel, spade, trowel, measuring cup or graduated cylinder, field scale, bucket auger, post hole auger, homogenization container w/ mixing tool, bucket, rinse bottle, purified water, paper towels

Sample Containers – Ziploc™ baggies, glass jars, labels, clear tape, pens, permanent marker, cooler(s), ice, thermometer

See Appendix A for a detailed list of soil sampling equipment.

## 10.0 STANDARDS AND REAGENTS

Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP# FLD-02 and the site-specific work plan.

## 11.0 PROCEDURES

### 11.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.
2. Obtain the necessary sampling and monitoring equipment.
3. Decontaminate or pre-clean equipment, and ensure that it is in working order.
4. Prepare schedules, and coordinate with staff, client, and regulatory agencies where necessary. It is also important to obtain access agreements if sampling is to occur on private property.
5. Perform a general site survey prior to site entry in accordance with the site-specific HASP.
6. Use stakes, flagging, or buoys to identify and mark all sampling locations followed by a GPS point (see GPS Trimble® GeoXT 2008 series SOP FLD 07.00). Specific site factors, including extent and nature of contaminant should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner



prior to soil sampling. It is the responsibility of the sampler to verify with the property owner that utility lines have been marked. If there is no property owner and there is concern for underground utility lines, it is the responsibility of the samplers to contact the state agency or contractor that can provide a marking service.

## **11.2 Sample Collection**

In general, there are two primary ways to collect a soil sample. Composite sampling involves taking several subsamples from a designated sample location and consolidating into one larger sample. Discrete sampling is defined as taking one sample from a single location. Composite and discrete sampling can be achieved by the sample techniques listed below.

### **11.2.1 Surface Soil Samples**

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Remove surface material to the required depth and use a stainless steel or plastic scoop to collect the sample.

This method can be used in most soil types but is limited to sampling near-surface areas. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A stainless steel scoop, lab spoon, or plastic spoon will suffice in most other applications. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required. Care should be exercised to avoid use of devices plated with chrome or other materials. Plating is particularly common with garden implements such as potting trowels. There are four depth classes that are typically used in Region 8: 0-2" range, 0-6" range, 6-12" range, and 12-18" range. The 0-2" and 0-6" range can usually be sampled with one of the tools listed above, but the deeper ranges generally require the use of one of the tools described in sections 11.2.2 and 11.2.3.

The following procedure is used to collect surface soil samples:

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.
3. If volatile organic analysis is to be performed, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the

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caps tightly.

4. Due to data quality requirements, some soil sampling events may require that each sub-sample of a composite be measured. This can be achieved two ways: by mass or by volume. Due to the remote nature of the sites in Region 8, it is recommended that composite samples are measured by volume. This requires the use of a measuring cup or graduated cylinder (of appropriate composition), placing the material into the measuring device to the desired volume, then adding the sub-samples to a larger sample container (plastic baggie for metals, glass jar for organics). Overall volume of sample will be dictated by analytical requirements.

### **11.2.2 Sampling at Depth with Augers and Thin Wall Tube Samplers**

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix B). The auger is used to bore a hole to a desired sampling depth and is then withdrawn from the hole. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The sampling assembly is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler.

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better suited for direct sample recovery since they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the auger flights. The continuous flight augers are satisfactory for use when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of three feet.

The following procedure is used for collecting soil samples with the auger:

1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod.
2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.
3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.
4. After reaching the desired depth, slowly and carefully remove the auger from the boring. When sampling directly from the auger, collect the sample after the auger is removed from the boring and proceed to Step 10.
5. Remove the auger tip from drill rods and replace with a pre-cleaned thin wall tube

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sampler. Install the proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring as the vibrations may cause the boring walls to collapse.
7. Remove the tube sampler, and unscrew the drill rods.
8. Remove the cutting tip and the core from the device.
9. Discard the top of the core (approximately 1 inch), as this possibly represents borehole debris material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.
10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
12. Abandon the hole according to applicable State regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

### **11.2.3 Sampling at Depth with a Trier**

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth. The following procedure will be used to collect soil samples with a sampling trier:

1. Insert the trier (Figure 2, Appendix B) into the material to be sampled at a 0 to 45 angle from horizontal. This orientation minimizes the spillage of sample.
2. Rotate the trier once or twice to cut a core of material.
3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. If volatile organic analysis is to be performed, transfer the sample into an appropriate,

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labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

#### **11.2.4 Sampling at Depth with a Split Spoon (Barrel) Sampler**

The procedure for split spoon sampling describes the collection and extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D 1586-67 (reapproved 1974). The following procedures will be used for collecting soil samples with a split spoon:

1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top.
2. Place the sampler in a perpendicular position on the sample material.
3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece (past the full length of the sample barrel) or compression of the sample will result.
4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.
5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. However, in order to obtain the required sample volume, use of a larger barrel may be required.
6. Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.

#### **11.3 Sample Sieving**

Analytical methods may require that a sample be separated by particle size. A sieve is the most effective method of separating coarse and fine material from a soil sample. Sieving and random

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sampling is also an effective method for soil sample homogenization (Schumacher et al, 1990). The site-specific SAP should be consulted when deciding what particle size of a soil sample should be submitted for analysis. Note that saturated soil samples should not be sieved. Those samples must first be dried before processing. Sieving procedures:

- Place sample in appropriate sized sieve (dry samples are optimal; wet samples will stick to the grid of a sieve).
- Use a catch pan that is of a material that won't compromise the integrity of the sample.
- Place lid on the sieve and shake vigorously to separate particle sizes.
- Transfer desired sample fraction to labeled container.
- Decontaminate thoroughly with brushes and/or compressed air before use on next sample.

## 12.0 DATA RECORDS AND MANAGEMENT

Once collected, samples are preserved, labeled, and stored for transport. A COC must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site Identification
- Date sampled
- Sampler initials
- Time
- Analysis to be performed

In addition, soil characteristics may need to be documented when sampling. Below is a standardized list of soil characteristics and their corresponding Unified Soil Classification System identifiers.

- GW – well-graded gravels, gravel and sand mixtures, little or no fines
- GP – poorly graded gravels, gravel and sand mixtures, little or no fines
- GM – silty gravels, gravel, sand, silt mixtures
- GC – clayey gravels, gravel, sand, clay mixtures
- SW – well-graded sands, little or no fines
- SP – poorly-graded sands, little or no fines
- SM – silty sands, sand-silt mixtures
- SC – clayey sands, sand-clay mixtures
- ML – inorganic silts and very fine sands
- CL – inorganic clays of low to medium plasticity
- OL – organic silts and organic silty clays
- MH – inorganic silts, micaceous or diatomaceous fine sandy or silty soils
- CH – inorganic clays of high plasticity
- OH – organic clays of medium to high plasticity, organic silts
- Pt – highly organic soils

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### **13.0 QUALITY CONTROL AND ASSURANCE**

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. An instruction manual for the operation of the hand auger equipment is provided in Appendix C of this SOP. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.
3. Always consult the SAP for duplicate sample frequency requirements.
4. Document any deviations from SOP's, work plan, SAP/QAPP, etc.

### **14.0 REFERENCES**

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

Mason, B.J., Preparation of Soil Sampling Protocol: Technique and Strategies. 1983 EPA-600/4-83-020.

Barth, D.S. and B.J. Mason, Soil Sampling Quality Assurance User's Guide. 1984 EPA-600/4-84-043.

U.S. EPA. Characterization of Hazardous Waste Sites. A Methods Manual: Volume II. Available Sampling Methods, Second Edition. 1984 EPA-600/4-84-076.

de Vera, E.R., B.P. Simmons, R.D. Stephen, and D.L. Storm. Samplers and Sampling Procedures for Hazardous Waste Streams. 1980 EPA-600/2-80-018.

ASTM D 1586-67 (reapproved 1974), ASTM Committee on Standards, Philadelphia, PA.

U.S. EPA, Environmental Response Team, standard operating procedures—Soil Sampling: U.S. Environmental Protection Agency, 1994, SOP #2012

Shumacher, B.A, Shines, K.C., Burton J.V., and Papp, M.L. (1990) "A Comparison of Soil Sample Homogenization Techniques."


## **EPA SOP FLD#-6 Shallow Stream Sediment Sampling**

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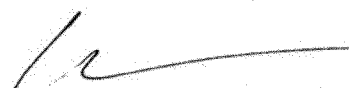
Replaces SOP: N/A

### Shallow Stream Sediment Sampling

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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.



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## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide field personnel a set of guidelines for the proper collection of stream sediment samples.

## 2.0 SCOPE AND APPLICABILITY

This SOP is applicable to the collection of shallow stream sediment samples. Analysis of sediment may be biological, chemical, or physical in nature and may be used to determine the following:

- Toxicity
- Biological availability and effects of contaminants
- Benthic biota
- Extent and magnitude of contamination
- Contaminant migration pathways and source
- Fate of contaminants
- Grain size distribution

The methodologies discussed in this SOP are applicable to the sampling of sediment in lotic environments. They are generic in nature and may be modified in whole or part to meet the handling and analytical requirements of the contaminants of concern, as well as the constraints presented by site conditions, equipment limitations and requirements of the site-specific Sampling and Analysis Plan (SAP). However, if modifications occur, they should be documented in field data sheet/field notebook and discussed in reports summarizing field activities and analytical results. For the purposes of this procedure, sediments are those mineral and organic materials situated beneath an aqueous layer in rivers and streams. Mention of trade names or commercial products for use in sediment sample collection does not constitute endorsement or recommendation for use.

## 3.0 SUMMARY OF METHOD

Sediment samples may be collected using a variety of methods and equipment, depending on the depth of the aqueous layer, the portion of the sediment profile required (surface vs. subsurface), the type of sample required (disturbed vs. undisturbed), contaminants present, and sediment type. Sediment is collected from beneath an aqueous layer directly, using a hand held device such as a shovel, trowel, or plastic scoop. Following collection, sediment is transferred from the sampling device to an appropriate sample container. If composite sampling techniques are employed, multiple grabs are placed into a container constructed of inert material, homogenized, and transferred to sample containers appropriate for the analyses requested.

*The homogenization procedure should not be used if sample analysis includes volatile organics.* In this case, if sediment is to be analyzed for volatile organics then the sample must be transferred to the appropriate sample container directly after collection. The sample bottle is filled completely and tapped lightly to get the trapped air out of the bottle. If the sediment settles in the bottle creating airspace then additional sediment should be collected. Repeat this step as many times necessary in order to have the sample bottle completely filled without having any air gaps.

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#### 4.0 ACRONYMS AND DEFINITIONS

COC	Chain of Custody
GPS	Global Positioning Systems
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	High-Density Polyethylene
OSHA	Occupational Health and Safety
QA	Quality Assurance
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
SOP	Standard Operating Procedure

Chain of Custody (COC): A chronological document that tracks movement of samples between entities from collection to disposal.

Composite Sampling: Sampling from several points or intervals and consolidating them into a larger sample.

Discrete Sampling: Sampling from a single location.

Global Positioning System (GPS): A geospatial referencing tool that is used for mapping and identification

Health and Safety Plan (HASP): A site specific document that identifies safety hazards and proper safety procedures. This normally includes hospital route maps and material safety data sheets.

Sampling and Analysis Plan (SAP): A site specific document that specifies events to take place in the field.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

Quality Assurance Project Plan (QAPP): A site specific document that specifies quality assurance activities and data quality objectives.

#### 5.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow United States Environmental Protection Agency (EPA), Occupational Safety and Health Agency (OSHA), and corporate health and safety procedures. More specifically, when sampling sediment from water bodies, physical hazards must be identified and adequate precautions must be taken to ensure the safety of the sampling team. The team member collecting the sample should not get too close to the edge of the water body, where bank failure may cause loss of balance. To prevent this, the person performing the sampling should be on a lifeline, and be wearing adequate protective equipment. If sampling from a vessel, appropriate protective measures and procedures must be implemented.

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## 6.0 CAUTIONS

Only collect sediment samples if it can be done so safely. Many unsafe conditions exist on streams and rivers. Also, review the SAP/QAPP or any other planning documents for analytical requirements and equipment selection. Consult the site HASP before performing any sample collection.

## 7.0 INTERFERENCES

Substrate particle size and organic matter content are a direct consequence of the flow characteristics of a water body. Contaminants are more likely to be concentrated in sediments typified by fine particle size and high organic matter content. This type of sediment is most likely to be collected from depositional zones. In contrast, coarse sediments with low organic matter content do not typically concentrate pollutants and are generally found in erosion zones.

## 8.0 PERSONNEL QUALIFICATIONS

All personnel who participate in field activities are required to obtain clearance in three mandatory health and safety programs: medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. In addition to this, any personnel who will participate in sediment sampling activities must read, understand, and sign the site specific HASP and the associated SAP/QAPP. Additionally, field personnel would benefit from understanding relevant SOPs including Sampling Equipment Decontamination SOP FLD 02.00, Sample and Labeling SOP FLD 11.00, and the General Field Sampling Protocols SOP FLD 12.00.

## 9.0 EQUIPMENT AND SUPPLIES

Equipment needed for collection of sediment samples may include:

Health and Safety Plan (HASP) - Personal floatation device, life line, neoprene waders/gloves, proper footwear, safety glasses, insulating clothing for cold water, etc.

Mapping & Location Tools - GPS units, site/local area maps, tape measure, compass, survey stakes, pin flags, camera, and 2-way radios.

Documentation - Field log book, field data sheet, chain of custody (COC), labels & clear tape, pens/sharpie, waterproof paper.

Sampling Tools - Plastic or other appropriate composition scoop, shovel, spade, trowel, homogenization container with mixing tool, rinse bottle, purified water, and paper towels.

Sample Containers -High-density polyethylene (HDPE) or other appropriate composition containers (50 mL and 1 liter [bulk] are frequently used), amber glass jars (organics analysis) labels, clear tape, pens, permanent marker, sealable plastic bags, cooler(s), and ice.

See Table 9.0-1 for a detailed list of sediment sampling equipment.

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## **10.0 STANDARDS AND REAGENTS**

Reagents are not used for preservation of sediment samples.

## **11.0 PROCEDURES**

### **11.1 Sample Preservation, Containers, Handling and Storage**

Chemical preservation of solids is not recommended. Cooling to 4 degrees Celsius (°C) is recommended for sediment samples. HDPE containers with Teflon™ lined caps are typically used for sediment samples. Sample container size is typically 50 milliliter (mL) for metal analysis and 1 liter for sediment toxicity testing. However, the sample volume is a function of the analytical requirements and will be specified in the SAP/QAPP. If analysis of sediment from a discrete depth or location is desired, sediment is transferred directly from the sampling device to a labeled sample container(s) of appropriate size and construction for the analyses requested. Transfer is accomplished with a decontaminated stainless steel or plastic lab spoon or equivalent.

If composite sampling techniques or multiple grabs are employed, equal portions of sediment from each location are deposited into a stainless steel, plastic, or other appropriate composition containers. The sediment is then homogenized thoroughly, to obtain a composite sample that is representative of the area and is then transferred to a labeled container. Transfer of sediment is accomplished with a stainless steel or plastic lab scoop or equivalent. Samples for volatile organic analysis must be transferred directly from the sample collection. It is important that when collecting sediment for volatile organic compounds analysis, the sample container is filled completely full and the sample container is tapped lightly to ensure all air is purged from the sample. This is done to minimize loss of contaminant due to volatilization.

All sampling devices should be decontaminated following procedures described in the Sample Equipment Decontamination SOP FLD 02.00. The sampling device should remain in its wrapping until it is needed. Each sampling device should be used for only one sample. Although disposable sampling devices for sediment are generally impractical due to cost and the large number of sediment samples which may be required, such devices may prove efficient and effective for difficult terrain/remote locations. Sampling devices should be cleaned in the field using the decontamination procedure described in the Sampling Equipment Decontamination SOP FLD 02.00.

### **11.2 Preparation**

Determine the objective(s) and extent of the sampling effort. Obtain access to private property if sample locations are located within private boundaries. The sampling methods to be employed, and the types and amounts of equipment and supplies required will be a function of site characteristics and objectives specified in the SAP and QAPP.

- Obtain the necessary sampling and monitoring equipment.
- Prepare schedules, and coordinate with staff, client, and regulatory agencies where appropriate.
- Decontaminate or pre-clean equipment, and ensure that it is in working order.

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- Perform a general site survey prior to site entry in accordance with the site-specific HASP.
- Use stakes, flagging, or buoys in addition to using a GPS (Refer to SOP FLD 07.00) to identify and mark all sampling locations. Specific site factors including flow regime, basin morphology, sediment characteristics, depth of overlying aqueous layer, contaminant source, and extent and nature of contamination should be considered when selecting sample locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

### 11.3 Sample Collection

Selection of a sampling device is most often contingent upon the depth of water at the sampling location and the physical characteristics of the sediment to be sampled. The following procedure consists of sampling surface sediment with a scoop, trowel or shovel from beneath a shallow aqueous layer:

For the purpose of this method, surface sediment is considered to range from 0 to 1 inch in depth and a shallow aqueous layer is considered to range from 0 to 12 inches in depth. Collection of surface sediment from beneath a shallow aqueous layer can be accomplished with tools such as spades, shovels, trowels, and scoops. Although this method can be used to collect both unconsolidated/consolidated sediment, it is limited somewhat by the depth and movement of the aqueous layer. Deep and rapidly flowing water may render this method less accurate than other methods such as utilizing a handheld dredge or coring device. However, representative samples can be collected with this procedure in shallow sluggish water provided care is demonstrated by the sample team lead. A plastic scoop will suffice in most applications. Care should be exercised to avoid the use of devices plated with chrome or other materials.

The following procedure will be used to collect sediment with a scoop, shovel, or trowel:

1. Using a decontaminated sampling implement, collect the desired thickness and volume of sediment from the sampling area.
2. Transfer the sample into an appropriate sample or homogenization container. Ensure that non-dedicated containers have been adequately decontaminated.
3. Surface water should be decanted from the sample or homogenization container prior to sealing or transfer; care should be taken to retain the fine sediment fraction during this procedure.

#### 11.3.1 Composite Sampling

Composite sampling consists of taking several sub-samples from a location and consolidating them into a larger sample. If data quality objectives dictate that each sub-sample of a composite be measured, it can be done two ways; by mass or by volume. For remote site field sampling activities (such as ones that typically occur in Region 8), it is

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recommended that sub-samples be measured by volume. This can be done with a graduated beaker/measuring cup or cylinder. Place the sub-sample in the measuring device, record the measurement, and transfer the sub-sample into a larger container where the complete composite sample will be processed.

Composite representative sample collection can also be accomplished without measurement of sub-samples. For sediment collection that does not require sub-sample measurement, larger amounts of sample can be collected in areas where sediment is more readily available. This method is used very frequently in high-gradient streams such as those found in the region.

### **11.3.2 Discrete Sampling**

Discrete sampling consists of taking a sample from a single location. This method requires that the selected location for a sediment sample have sufficient amount of material for the analytical requirements. In general, sediment samples in a stream are difficult to obtain from a single location; therefore composite samples are more commonly collected.

## **12.0 DATA RECORDS AND MANAGEMENT**

Once collected, samples are labeled and stored for transport (at 4°C). A COC must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site Identification
- Date sampled
- Sampler initials
- Time
- Analysis to be performed

## **13.0 QUALITY CONTROL AND ASSURANCE**

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the SAP and QAPP. Equipment inspection must occur prior to sampling, and they must be documented.
3. QA samples should be sampled at a standardized frequency. Field duplicates are generally sampled at a rate of 1:20.

## **14.0 REFERENCES**

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

Mason, B.J., Preparation of Soil Sampling Protocol: Technique and Strategies. 1983 EPA-600/4-83-020.

Barth, D.S. and B.J. Mason, Soil Sampling Quality Assurance User's Guide. 1984 EPA-600/4-84-043.

U.S. EPA. Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. 1984 EPA600/4-84-076.

de Vera, E.R., B.P. Simmons, R.D. Stephen, and D.L. Storm. Samplers and Sampling Procedures for Hazardous Waste Streams. 1980 EPA-600/2-80-018.

U.S. EPA, Environmental Response Team, standard operating procedures—Sediment Sampling: U.S. Environmental Protection Agency, 1994, SOP #2016.



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**Table 9.0-1: Sediment Sampling Equipment**

<u>Category</u>	<u>Item</u>	<u>Use</u>	<u>Comment</u>
Health and Safety	Gloves	Protection from absorption of contaminants	Nitrile or neoprene are recommended
Health and Safety	Waders	Slip/contaminant protection	Any type are acceptable
Health and Safety	Safety Glasses	Eye protection	Sunglasses for UV protection
Health and Safety	Layered Clothing	Protection from hypothermia	Polyester base layers only
Mapping/Location	GPS unit	Sample station locating	Pre-loaded with site locations
Mapping/Location	Maps	Location identification	Must contain most current information
Mapping/Location	Two-way radios	Communication	Extra batteries or charger required
Documentation	Field Logbook	Site data and conditions documentation	Waterproof pages
Documentation	Chain of Custody	Sample handling/identification	Pre-printed using Scribe
Documentation	Labels	Sample identification	Pre-printed using Scribe
Documentation	Clear tape & Scissors	Label protection	
Sampling Tools	Bucket/Transfer Device	Sample transfer (if required)	Can also be used for sample homogenization
Sampling Tools	Sediment Scoop	Sediment sampling	Select based on analysis
Sampling Tools	Cooler	Sample containment	Cool to 4°C
Sampling Containers	Amber glass jars	Volatile Organics Analysis sample containment	Tight cap seal
Sampling Containers	50 ml HDPE (widemouth)	Metals analysis sample containment	
Reagents	10% Nitric Acid (HNO <sub>3</sub> ) solution	For decontamination of metals sampling equipment	Pre-mixed at lab
Reagents	10% Hydrochloric Acid (HCl) solution	For decontamination of organics sampling equipment	Pre-mixed at lab


## **EPA SOP FLD#-10 Pore Water Sampling**

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### Pore Water Sampling

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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures, methods, and considerations to be used when obtaining a pore water sample using PushPoint® samplers.

## 2.0 SCOPE AND APPLICABILITY

This document describes procedures for pore water sampling using PushPoint® samplers and is based on the Operators Manual and Applications Guide provided by MHE Products (Ver. 2.01,2/15). It is intended to be used by field personnel when collecting and handling samples in the field. All deviations from this SOP must be noted in the site-dedicated field logbook.

## 3.0 SUMMARY OF METHOD

Sediment pore water is collected using a pore water extractor, called a PushPoint® (Figure 1) which is made out of stainless steel tubing developed by MHE Products. The sampling end of the PushPoint® is inserted into the sediment to the desired depth, and pore water is extracted using a syringe or peristaltic pump.

## 4.0 ACRONYMS AND DEFINITIONS

COC	Chain of Custody
DOC	Dissolved Organic Compounds
DOT	United States Department of Transportation
DQO	Data Quality Objective
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
IATA	International Air Transportation Association
OSHA	Occupational Safety and Health Administration
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SVOC	Semi-Volatile Organic Compound
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

Health and Safety Plan (HASP): A site-specific document outlining potential safety hazards and hazard mitigation techniques.

Occupational Safety and Health Administration (OSHA): An agency that regulates health and safety standards in the United States.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

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## **5.0 HEALTH AND SAFETY**

Proper safety precautions must be observed when collecting pore water samples. Refer to Environmental Services Assistance Team (ESAT) site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When following this SOP, minimize exposure to potential health hazards in the field by using personal protective equipment (protective clothing, eye wear and gloves). Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

## **6.0 SAMPLE MANAGEMENT**

The following precautions should be considered when collecting pore-water samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party (see SOP FLD-11.00, or current version, "Sample Custody and Labeling").
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples must conform to all United States Department of Transportation (DOT) and/or International Air Transportation Association (IATA) hazardous materials shipping requirements.
- Documentation of field sampling is done in a bound logbook.
- Chain of custody (COC) documents must be filled out and remain with the samples until custody is relinquished until analysis is complete (or samples are disposed).
- All shipping documents, such as bills of lading, etc., shall be retained by the project leader and stored in a secure place.

## **7.0 INTERFERENCES**

The following sections describe potential interferences when sampling for trace level contaminants. For decontamination procedures, see the Sampling Equipment Decontamination SOP FLD 02.00.

### **7.1 Potential Volatile Organic Analysis (VOA) Sampling Interferences**

Pore water samples for volatile organic compound (VOC) and semi-volatile organic compounds (SVOC) analysis must be collected in 40-ml amber glass vials with Teflon<sup>®</sup> septa. The vials may be preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two week holding time, whereas, unpreserved samples have only a seven day

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holding time. Normally, either preserved or unpreserved vials can be used, but there are instances where the use of unpreserved vials is preferred. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

Samples for VOC and SVOC analysis must be collected using either stainless steel or Teflon<sup>®</sup> equipment. Samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial, and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be refilled. Care should be taken not to flush any preservative out of the vial during topping off. If bubbles are still present after attempting to refill and cap the vial, a new vial should be obtained and the sample re-collected.

## **7.2 Potential Dissolved Metals or Dissolved Organic Compound Sampling Interferences**

If a dissolved metals or Dissolved Organic Compounds (DOC) pore water sample is to be collected, in-line filtration or post-collection filtrations are acceptable approaches. The in-line filter apparatus uses disposable, high capacity filter cartridges (barrel-type) or membrane to filter the sample. The high capacity, barrel-type filter works well due to the higher surface area associated with this configuration. Post-collection filtration involves two approaches. The first approach is to take the sample water and filter it through a .45 micron filter apparatus. The second approach involves the use of a syringe with a .45 micron acrodisc filter attached to end of syringe.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations, including filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloiddally-associated trace elements, and concentration of organic matter. Therefore, consistency of sample technique and filter characteristics is critical in the comparison of short-term and long-term results.

## **7.3 Special Precautions for Trace Contaminant Pore Water Sampling**

1. A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled, and the gloves should be donned prior to handling sampling equipment and sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
2. Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately from samples suspected of only having trace levels of contaminants.

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3. All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
4. Samplers must use new, verified, certified clean disposable equipment, or pre-cleaned non-disposable equipment.

## 8.0 PERSONNEL QUALIFICATIONS

Any personnel involved with field sampling activities must be cleared for health and safety. Clearance includes medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. Personnel who will be collecting pore water samples must be familiar with this SOP and any other relevant SOPs, including the Sample Equipment Decontamination SOP FLD 02.00, Sample Preservation SOP FLD 03.00, Water Quality Measurements with the In-Situ® Multi-Parameter Meter SOP FLD 09.00, Sample and Labeling SOP FLD 11.00, and General Field Sampling Protocols SOP FLD 12.00.

## 9.0 EQUIPMENT AND SUPPLIES

The PushPoint® sampler consists of a tubular 3/16 stainless steel body with a screened zone at one end and a sampling port at the other. It comes with a guard rod that is nested in the tube during deployment in order to provide structural support and to prevent plugging and deformation of the screened zone (Figure 1). The screened zone consists of a series of interlaced machined slots that form a short screened zone with approximately 20% open area. Additional filters can be placed over the screened zone if additional screening is needed. Pore water is collected through the opposite end of the PushPoint® sampler through peristaltic flexible tubing using a syringe through the sampling port. Tygon® tubing is the preferred tubing to be used with PushPoint® samplers. However, other tubing can be used, if allowed by data quality objectives (DQOs) for the specific application. PushPoint® samplers can be custom made to any width or length.

There are many modifications that can be incorporated into the procedure to satisfy DQOs for a specific application. The procedures discussed in the following sections provide guidance on the basic operation of the PushPoint® and issues to consider when deploying the PushPoint® sampler to collect pore water.

Other equipment used in the process of pore water sample collection includes syringes, flanges, tubing, sample bottles or containers, and filters.

## 10.0 STANDARDS AND REAGENTS

Reagents will be used for preserving samples and for decontaminating sampling equipment (refer to the Sampling Equipment Decontamination SOP FLD 02.00 and Sample Preservation SOP FLD 03.00). The preservatives required are determined by the analysis to be performed and will be specified in the Sampling and Analysis Plan (SAP)/Quality Assurance Project Plan (QAPP), but usually include nitric acid (total recoverable and dissolved metals samples), hydrochloric acid (VOC samples), and phosphoric acid (DOC samples). The sampler should also be aware of any special sampling considerations,



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contamination issues, and sample compositing and mixing methods that could affect their sampling efforts. Appropriate regional guidance and procedures should be consulted for detailed sample collection, preservation, handling and storing, equipment decontamination, and quality assurance/quality control (QA/QC) procedures. The sampler should preserve and immediately cool all water samples to 4°C ( $\pm 2^\circ\text{C}$ ) upon collection and samples should remain cooled until the time of analysis (do not freeze water samples).

## 11.0 PROCEDURES

It is critical in the collection of pore water to avoid surface water intrusion. Water will flow in a path of least resistance. If space is created around the sides of the PushPoint® sampler during deployment, surface water may flow down the outside of the tube to the screened area and into the intended sample. Therefore, the PushPoint® can be used with a sampling platform or flange (Figure 2), especially when collecting pore water near the sediment-surface water interface. However, if pore water is collected from deep in the sediments or in cobble-bottom streams, a flange may not be necessary. Additionally, it is important to note that a platform is only useful in specific situations when you are sampling multiple holes and specific depths and when sampling at shallow depths where the integrity of the hole may be a concern. It is critically important to collect samples from the hyporheic zone, or the area beneath the streambed where shallow groundwater is mixed with surface water (this area is critical to benthic macro-invertebrates and fish spawning activity). When inserted through the sampling platform, or flange, the flange should fit securely around the PushPoint® to eliminate surface water intrusion from around the PushPoint® body during sample collection.

The flange can be made of any material that will not cross contaminate the intended sample. If full scan analytical analysis is required, the flange should be made of inert material such as stainless steel or Teflon®. The size of the flange depends on the volume of pore water to be collected. If large volumes of pore water are to be collected, use a large flange size. If it is not practical to use a large flange, then multiple PushPoints® with smaller flanges can be deployed and smaller volumes can be collected from several PushPoints® for a composite sample. If multiple PushPoints® are deployed, they should be spaced at least 30 cm apart.

### 11.1 PushPoint® Sampler Basic Operation

The PushPoint® sampler should be inserted into the sediment as carefully as possible (Figure 2). When deploying the PushPoint®, care must be taken not to disturb the sampling area. If the sampler is wading in the water body, the sampler should lean out and insert the PushPoint® as far as possible away from where he/she is standing in order to reduce potential effects of the sampler on the integrity of the pore water sample. Depth of penetration of the PushPoint® into the sediment depends on the objectives of the specific investigation. Once depth is established for sample collection, be sure to measure and record the sampling depth in the logbook.

After the PushPoint® has been deployed, carefully remove the guard rod and attach the sample tubing (Figure 3). The other end of the sample tubing can be connected to the sample withdrawing device, such as a peristaltic pump or syringe (Figure 4). Before collecting a pore water sample, be sure to purge out all air and surface water from the PushPoint® sampler and sample tubing with the appropriate amount of pore water. At least three volumes of pore water (until water is clear) should be purged before sample collection.

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## **11.2 Peristaltic Pump/Vacuum Jug Collection**

The peristaltic pump/vacuum jug can be used for sample collection because it allows for sample collection without the sample coming in contact with the pump head tubing. This is accomplished by placing a Teflon® transfer cap assembly onto the neck of a clean standard 1-liter amber glass container. Teflon® tubing (3-inch outside diameter) connects the container to both the pump and the sample source. The pump creates a vacuum in the container, thereby drawing the sample into the container without it coming into contact with the pump head tubing.

Because the sample is exposed to a vacuum and is agitated as it enters the vacuum jug, this method cannot be used for collection of VOC samples. An alternative method for collecting VOC samples involves filling the Teflon® tubing with sample by running the pump for a short period of time. Once the tubing is full of water, the tubing is removed from the PushPoint® and, after the tubing is disconnected from the pump head tubing, the water is allowed to drain, by gravity, into the sample vials. Alternatively, without disconnecting the tubing from the pump head, the contained sample can be pushed out of the tubing and into the sample vials by reversing the peristaltic pump at low speed.

For samples that are collected for metals analyses, or other analysis not affected by the silastic tubing, it is permissible to collect the sample directly from the discharge of the pump head tubing after an adequate purge has been completed. When collecting samples in this manner, there are several considerations to be aware of. The pump head tubing (silastic, etc.) must be changed after each sample and a rinsate blank must be collected from a representative piece of the pump head tubing (only one blank per investigation). Also, precautions must be taken to ensure that the end of the discharge tubing is not allowed to touch the ground, or other surface, in order to maintain the integrity of the sample when it is collected in this manner.

## **11.3 Syringe**

Syringes, in conjunction with PushPoint® samplers, can be used to collect pore water samples if the integrity of the sample analysis will not be compromised. The tubing from the sampling port of the PushPoint® can be directly attached to a syringe and a pore water sample can be manually withdrawn from the sediment. The syringe can be used as the final sample container or the pore water can be transferred to another container, depending on project objectives and analytical requirements.

## **11.4 Sample Handling and Preservation Requirements**

1. Pore water will typically be collected from sediments using a PushPoint® and placed directly into the sampling containers. A syringe may then be used to transfer the sample from the sampling container into the appropriate container.
2. When transferring the pore water sample from a collection device, make sure that the device does not come in contact with the final sample containers. The syringe used in the sample transfer is the only piece of equipment that should be in contact with the transfer vessel and the final sample container.
3. Place the sample into the appropriate labeled container. Samples collected for VOC

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analysis must not have any headspace (see Section 7.1). All other sample containers must be filled with an allowance for ullage.

4. All samples requiring preservation must be preserved as soon as practically possible after sample collection. If preserved VOA vials are used, these will be preserved with concentrated hydrochloric acid prior to departure for the field investigation. All other chemical preservatives required for the remaining suite of analytes will be specified in the site-specific SAP. The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not acceptably preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples will be specified in the site-specific SAP/QAPP and the Sample Preservation SOP FLD 03.00.

## 12.0 DATA RECORDS AND MANAGEMENT

Once collected, samples are preserved, labeled, and stored for transport. A chain of custody form must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site identification
- Date sampled
- Location identification
- Sampler initials
- Time
- Analysis to be performed
- Preservative

Any other pertinent data should be recorded in the site dedicated field logbook.

## 13.0 QUALITY CONTROL AND ASSURANCE

The following general QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.
2. In general, concurrent (duplicate) sample collection at a frequency of 10% is required for most sampling activities. Field blanks at a frequency of one per day are also generally required. Consult the corresponding SAP/QAPP for specific QA/QC sampling frequency. Below is a list of usual pore water QA/QC sample types and the inaccuracy they are intended to detect:
  - Duplicate sample – two samples collected at the same location at the same time, intended to detect variability inherent in collection, processing, and handling procedures
  - Field blank – checks cross-contamination during sample collection, preservation, and shipment as well as in the laboratory.
  - Rinsate blank – detects equipment contamination due to inadequate decontamination

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procedures

3. All instrumentation should be operated in accordance with operating instructions as supplied by the manufacturer unless otherwise specified in the work plan or SAP/QAPP. Equipment calibration activities should be conducted and documented prior to sampling and/or operation of equipment.
4. Document any deviations from SOPs, work plan, SAP/QAPP, etc.

#### **14.0 REFERENCES**

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

MHE Products, PushPoint® Sampler (US Pat. # 6,470,967), Operators Manual and Applications Guide, Ver. 2.01, 2/15.

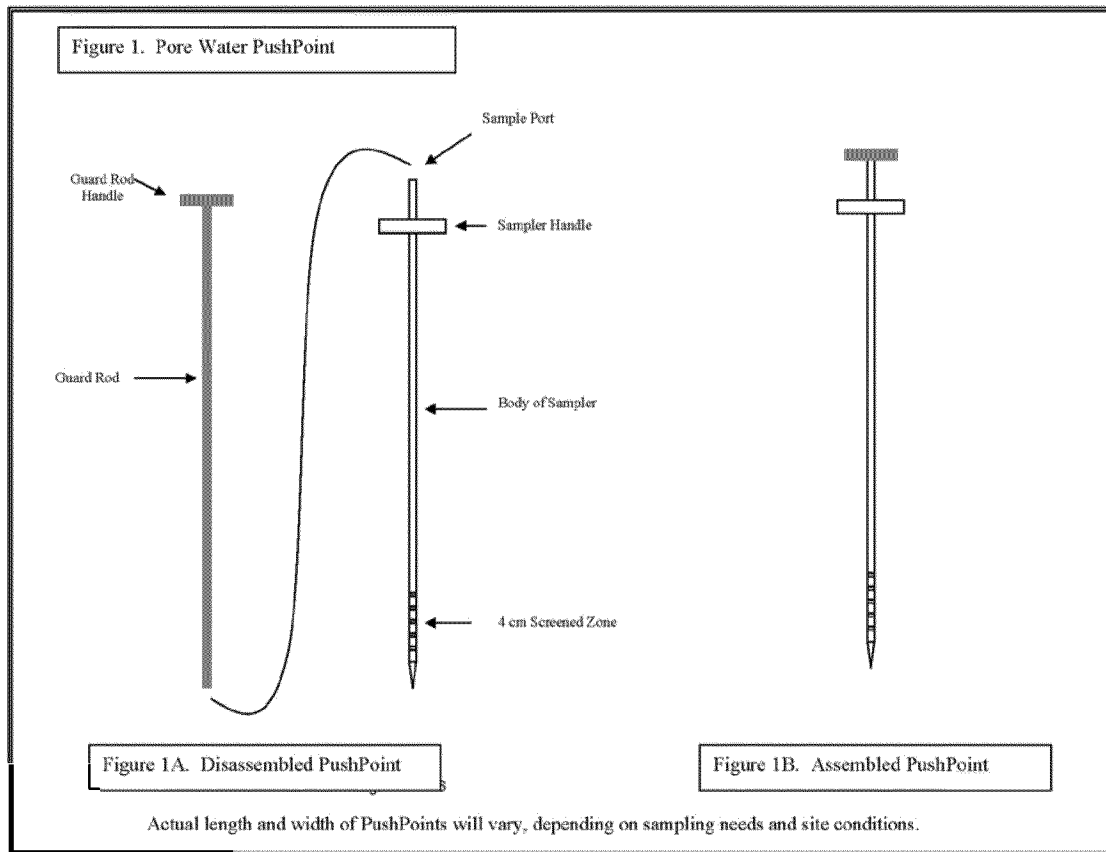
#### **15.0 FIGURES**

Figures 1-4 show equipment operation and basic sampling techniques.

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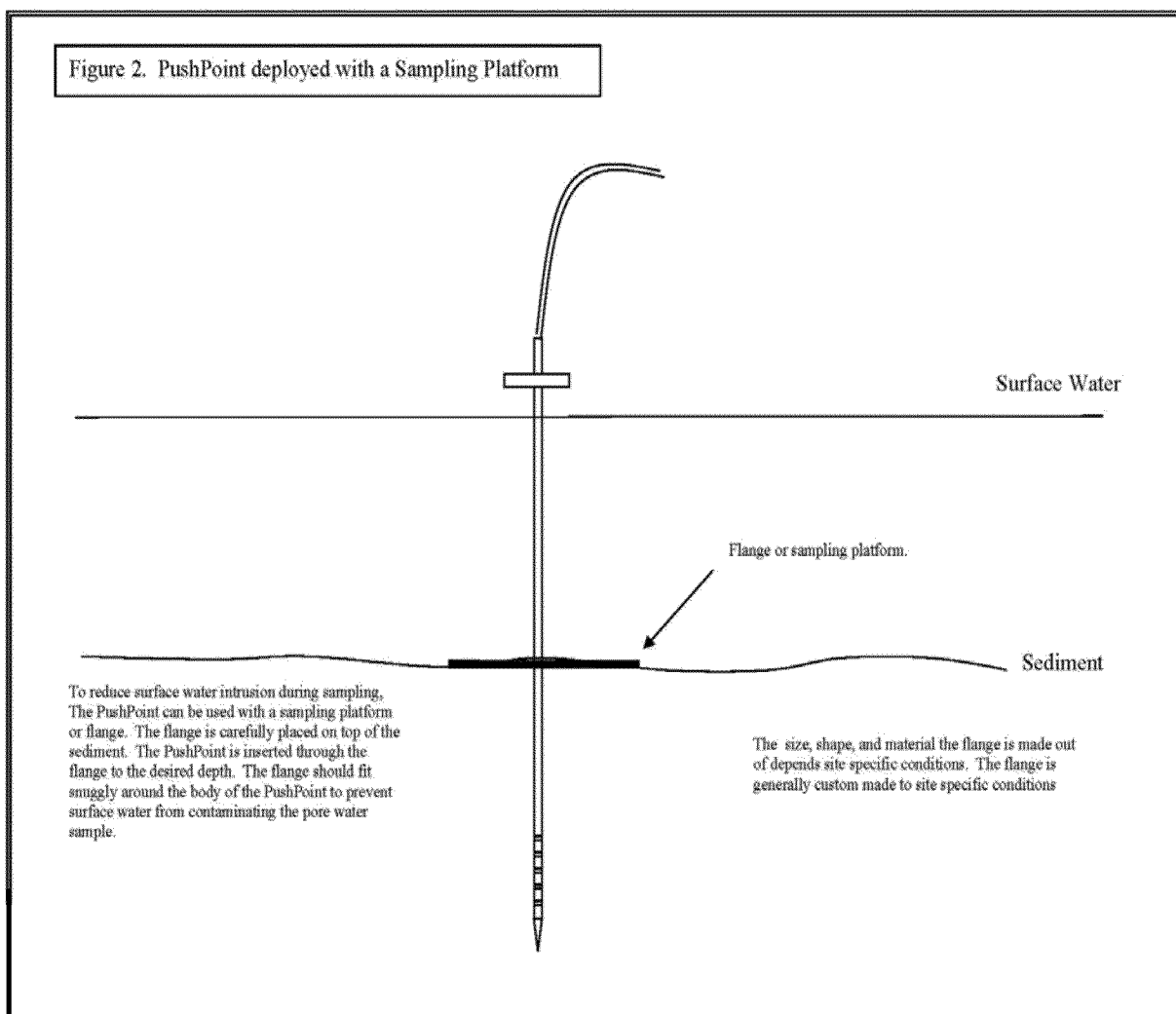
**Figure 1 - Pore Water PushPoint®**



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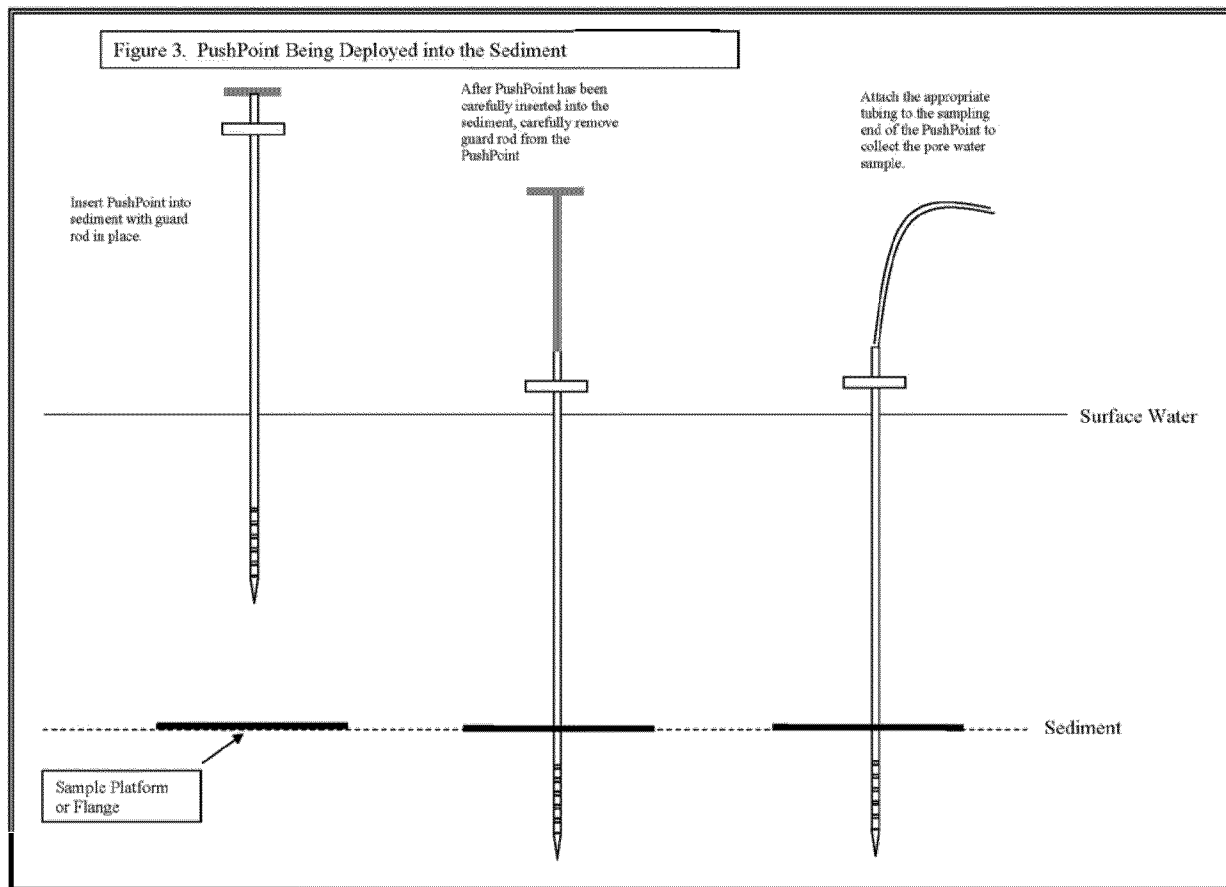
**Figure 2 - PushPoint® deployed with a Sampling Platform**



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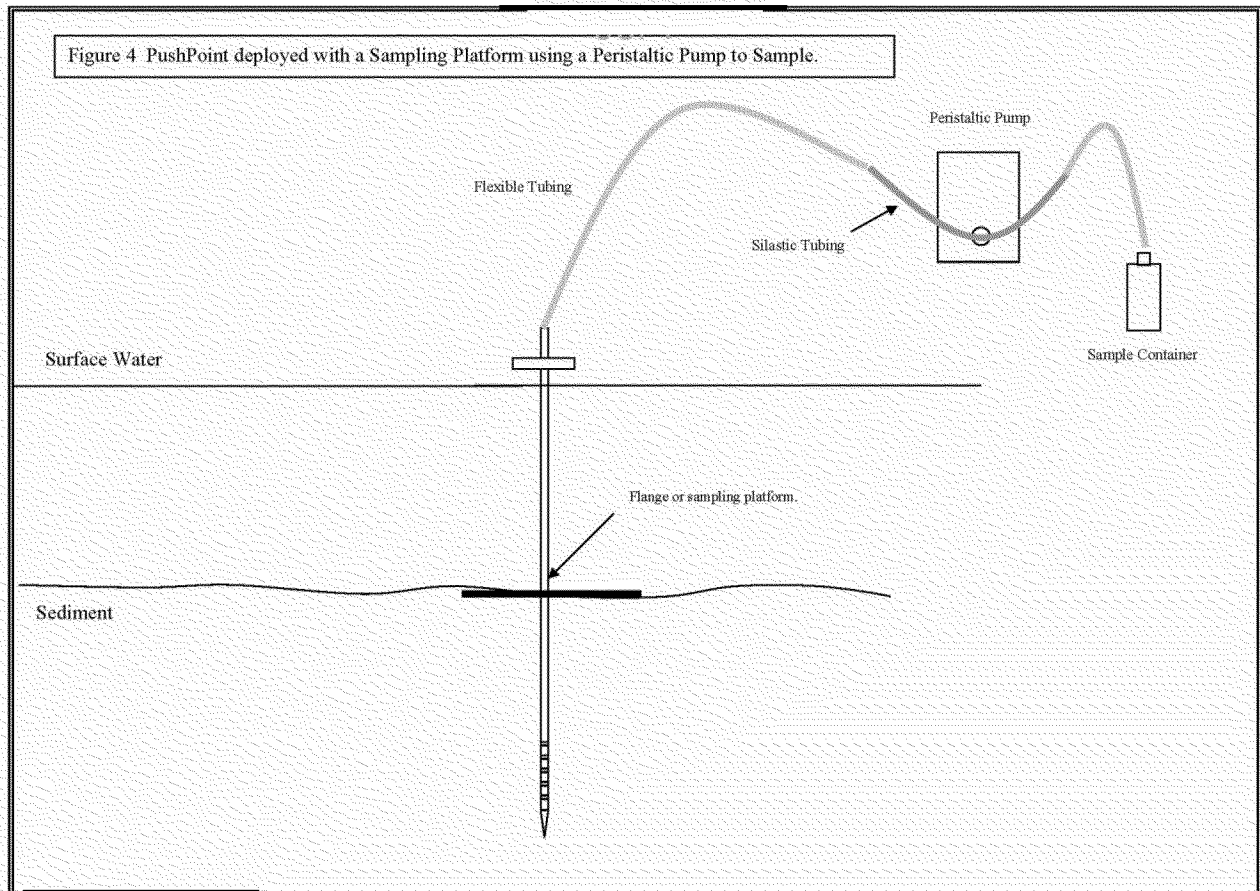
**Figure 3 - PushPoint® Being Deployed into the Sediment**



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**Figure 4 - PushPoint® deployed with a Sampling Platform using a Peristaltic Pump to Sample**





**Appendix C – HASP**  
**(To Be Provided at a Later Date)**